JULY, 1953

Journal of METALS

July JOURNAL OF METALS Highlights:

"Monolithic Linings in Furnace Spouts" - p. 877
"Open Hearth Slag Removed Economically by Blasting" - p. 881
"Electrical Resistance of Titanium Metal" - p. 903

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Open-Hearth Grade	50 to 55%	8%	3%
Foundry Grade	50 to 55%	approx. 10%	3%

Vanadium Oxide (3 Grades)	Composition		
	Vanadium Oxide (V_2O_5)	Sodium Oxide (Na_2O)	Calcium Oxide (CaO)
Fused	86 to 89%	approx. 10%	approx. 2%
Sodium Polyvanadate (Red Cake)	approx. 85%	approx. 9%	approx. 2%
High-Purity	approx. 99.5%	...	...

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Journal of METALS

VOL. 5, NO. 7 JULY, 1953

Published to provide a continuing, authoritative, and up-to-date record of technological, engineering, and economic progress in all branches of the metals industry by the Metals Branch American Institute of Mining and Metallurgical Engineers, Inc. 29 West 39th Street, New York 18

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COVER

In this electric furnace heat of extra low carbon stainless steel, Simplex ferrochrome pellets, product of Electro Metallurgical Co., a div. of Union Carbide & Carbon, have just been dumped on top of the bath. The pellets are quickly dissolved by the heat, thus saving furnace time. See article p. 886.

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Published the first day of each month by the American Institute of Mining and Metallurgical Engineers, Inc., 29 West 39th Street, New York 18, N. Y. Telephone: PEnnsylvania 6-9220. Subscription $8 per year for non-AIME members in United States and North, South and Central America; $10, foreign; $6 for AIME members, or $4 additional in combination with a subscription to "Mining Engineering" or "Journal of Petroleum Technology". Single copies, 75 cents; single copies foreign, $1.00; special issues, $1.50... The AIME is not responsible for any statement made or opinion expressed in its publications....

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Registered cable address, AIME New York... Indexed in Engineering Index, Industrial Arts Index and Chemical Abstracts... Reentered as second-class matter Sept. 21, 1949, at the post office at New York, N. Y., under the Act of March 3, 1879. Additional entry established at Manchester, N. H.

Meet The Authors

James L. Wyatt (p. 903) is an AIME Member belonging to the Cleveland Section. Dr. Wyatt was born at Williamsburg, Ky. He holds degrees from the University of Kentucky and Massachusetts Institute of Technology. Following graduation in 1947, he became associated with the titanium div., National Lead Co., as a development engineer working on titanium production. Since February 1953, he has been assistant technical manager for Horizons Inc., Cleveland. The title of one of his previous articles is *Electrical Resistivity of Titanium Slags*. Among his many hobbies are flying and photography, in addition to outdoor sports. Dr. Wyatt is also a member of the ASM.

Donald R. Mash (p. 937) is a native of Greensburg, Pa., now residing at Livermore, Calif. He holds B.S., M.S., and Ph.D. degrees from Stanford University and Carnegie Institute of Technology. For two years he was in the Army and following his discharge was a research metallurgist at the Los Alamos Atomic Energy Commission research project. In 1947 Dr. Mash became associated with the Columbia Steel Co., Pittsburgh, Calif., as dept. metallurgist, open hearths and rolling mills. For a time he was research assistant at Stanford University. Since 1952 he has been with the California Research & Development Co., as metallurgical engineer.

D. R. MASH

G. M. BURRIER

G. M. Burrier (p. 878) graduated from the University of Pennsylvania in 1932. Mr. Burrier began his career in the production dept. of Youngstown Pressed Steel Co. He then joined the Republic Steel Corp. as a third helper in the Youngstown plant. He held various jobs in open hearth, melting and was open hearth fuel engineer for a short period. In 1941 he was transferred to the South Chicago plant as assistant open hearth superintendent in both open hearth and electric furnace depts. From 1946 to 1950 he was superintendent of open hearth and then transferred to Cleveland. Here he became secretary of open hearth committee. In 1950 he accepted the position of assistant general manager of the Midland works, Crucible Steel Co.

D. J. Whitehead (p. 924) attended Manchester University, graduating in 1945 with the degree of B.Sc. From 1946 to 1948 he was assistant metallurgist for Richard Johnson & Nephew Ltd., Manchester. He is presently associated with Magnesium Elektron Ltd. as metallurgist, a position he has held since 1948. He is a member of the Institute of Metals and Iron and Steel Institute. Electronics is one of Mr. Whitehead's hobbies, as well as gardening and walking.

D. J. WHITEHEAD

G. R. HEFFERNAN

G. R. Heffernan (p. 884) had been a lieutenant in the Royal Canadian Engineers, following graduation from the University of Toronto in 1943 (Ba.Sc). After his discharge in 1945, Mr. Heffernan did graduate work in metallurgy at the University of British Columbia. During this time he was also an instructor in the dept. of metallurgy. He then became associated with the Westland Iron & Steel Foundries, Vancouver as metallurgist and production manager. He remained with this company until 1948 when he was named general superintendent of Vancouver Steel Co. Ltd. and Vancouver Rolling Mills Ltd., div. Western Canada Steel Ltd. Mr. Heffernan is a Member of AIME, Assn. of Professional Engineers of British Columbia, ASM, CIMM, and Electric Metal Makers Guild.

Lewis D. Hall (p. 937) was born at Portland, Ore. He attended the Universities of Nebraska and California and has B.S., M.S., and Ph.D. degrees. In 1949 he joined the staff of the Institute for the Study of Metals, University of Chicago. A year later Dr. Hall became associated with Stanford University as an instructor. In 1952 he was named an assistant professor, a position he now holds. His diversified hobbies include the study of foreign languages and playing tennis.

L. D. HALL

E.C.W. PERRYMAN

E. C. W. Perryman (p. 906) is associated with the Aluminum Laboratories, Ltd., Kingston, Ont. Born in Middlesex, England, Mr. Perryman received his M.A. from Magdalene College, Cambridge. For three years he was in the metallurgical div. of the Royal Aircraft Establishment. He joined the British Non-Ferrous Metals Research Assn. in 1946 and resigned in 1951 to accept a position with the Aluminum Laboratories. As his hobbies, Mr. Perryman counts bridge, tennis, and badminton. He has presented several papers before the Institute of Metals (England) and he also holds membership in the Institution of Metallurgists (England).

Robert Steinitz (p. 891) was born in Beuthen, Germany. Dr. Steinitz is a graduate of the Institute of Technology, Charlottenburg and the University of Goettingen, Germany. In 1933 he was employed by Fred Krupp AG and a few years later came to the United States. He joined the Crucible Steel Co., and then became associated with the American Electro Metal Corp., where he is research supervisor. A Member of AIME, he previously presented papers.

J. C. MacNeill (p. 879) is superintendent of No. 2 and 4 open hearths, Bethlehem Steel Co., Bethlehem, Pa. He has worked at Bethlehem since graduation from Yale (A.B.) in 1915. He began his career as third helper in open hearth and worked up to his present position. In his spare time Mr. MacNeill enjoys a few rounds of golf and does some photographic work.

J. B. Wagstaff (p. 895) has been with the U. S. Steel Research Laboratory since 1949 and is now engaged in fundamental research on blast furnaces. He was born in London and graduated from Sheffield University (B.Sc.) and Massachusetts Institute of Technology (M.S.). In 1937 Mr. Wagstaff joined Hadfields Ltd. as combustion engineer. He remained with this firm until 1944 when he became associated with the British Coal Utilization Research Assn. Prior to joining U. S. Steel, he was employed by Hydrocarbon Research, Inc., engaged in process design work on oxygen gasification of coal. Gardening and photography occupy his spare moments.

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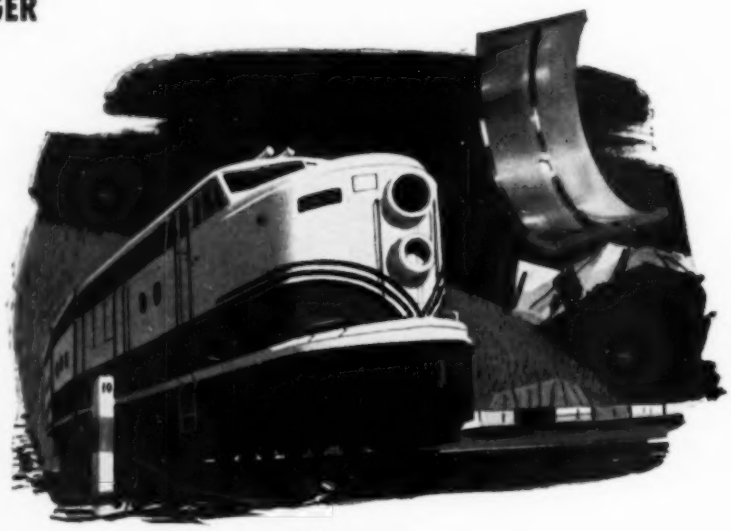

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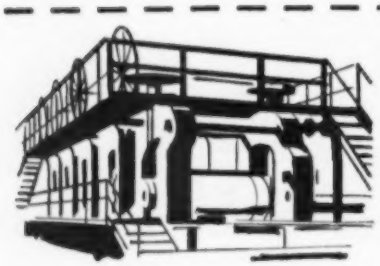
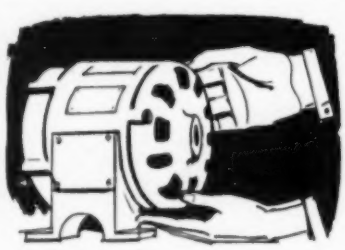
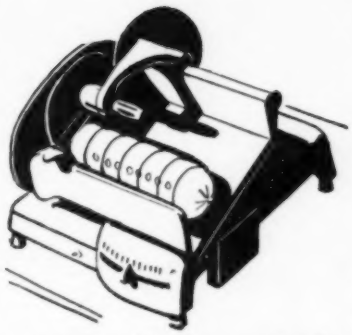


ROLLING MILLS now use aluminum bearings on the roll necks. Under these extreme pressures, aluminum bearings last far longer than those previously used.

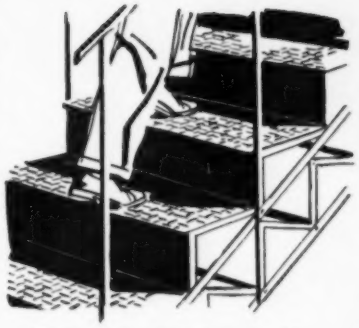


BUSHINGS for electric motors are now cast of Alcoa bearing alloy.

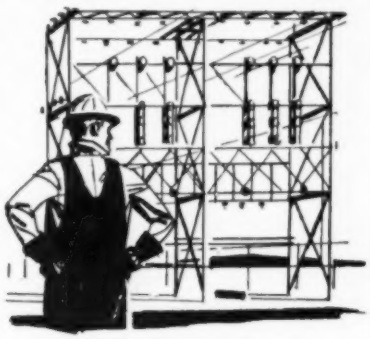
REFRIGERATION and air conditioning equipment is proving a big market for aluminum bearings—because of long life—easy machineability.



CASTINGS—Aluminum sand and permanent mold castings, like parts for this meat slicer, are strong and sound when Alcoa makes them. They compete in price with castings of any metal when you figure their cost machined, finished and completely assembled.



***TREAD PLATE**—Strong Alcoa Aluminum Tread Plate is widely used in refineries and chemical plants because it is nonsparking—in trucks and tractors because it is lightweight.



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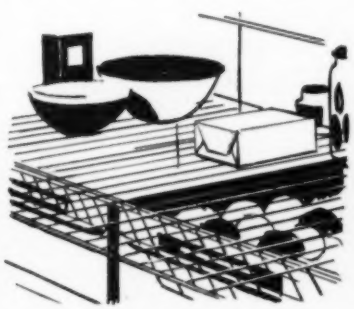
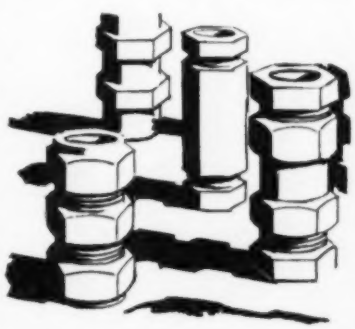


***WIRE**—Nonstaining, nontoxic aluminum wire is widely used for refrigerator shelves. Its bright, lustrous appearance lasts for years. You get three times more than other metals—pound for pound.



***SCREW MACHINE STOCK**—Aluminum Screw Machine Stock goes three times further per pound than heavy metals. It machines freely—won't rust—is widely used for fittings and fasteners.

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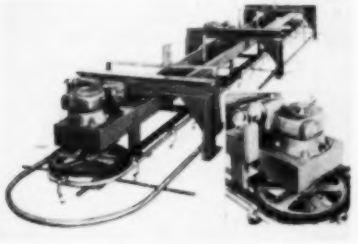
peratures to 200°F and a standard humidity cycle of 20 to 95 pct from +35°F to +185°F, has been introduced by *Murphy & Miller, Inc.* The units are made in five sizes with test space capacity from 4 to 36 cu ft.

2—MILD STEEL ELECTRODE: An improved E6-10 electrode for welding mild steel in all positions has been announced by *Air Reduction Sales Co.* It offers deep penetration without undercutting, a minimum of spatter, and uniform weld deposits without fingernailing.

3—PLATING EQUIPMENT: A packaged semiautomatic plating system to augment still tank plating for



electroplating has been designed by *Wagner Brothers, Inc.* When a run is completed, or between runs of a certain part, the system may be altered for a different job by shifting the pusher shoes to the desired spacing. Spacing to any dimension in 2-in. increments may be accomplished.

4—X-RAY UNIT: A portable industrial X-ray unit that is designed specifically for rapid inspection of welds, ship and aircraft equipment is available from *North American Philips Co., Inc.* The X-ray tube head also contains the high tension generator of 150,000 v output. Combination tube and generator unit has a diameter of 10⅝ in. and is 35 in. long. Power supply can be either 115 or 230 v ac. Current drain is 20 amp.

5—INDUSTRIAL TRUCK: A gas powered industrial truck with electric transmission has been introduced by *Automatic Transportation Co.* Capacities range from 4000 to 6000 lb. There is no mechanical connection between the engine and wheel so transmission shock loads to the engine are entirely eliminated.

6—CR-MN STAINLESS: An austenitic alternate for 18-8 stainless has been developed by *Allegheny Ludlum Steel Corp.* It is produced with manganese, chromium, and less than 1 pct nickel as principal alloying elements. In applications where critical requirements include the mechanical properties of welds, ductility, nonmagnetic qualities, and cold working to high strength, the chromium manganese steels should be considered.

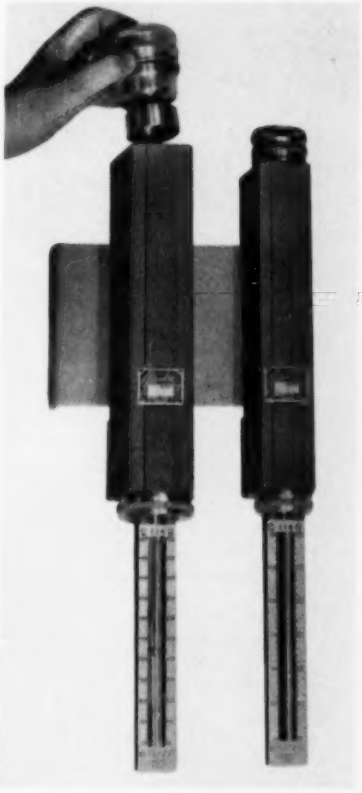
7—ELECTROPHORESIS-DIFFUSION: Double sensitivity by directing light through the cell and specimen twice is achieved by the new Spinco model H electrophoresis diffusion instrument offered by *Specialized Instruments Corp.* The apparatus is housed in a single laboratory cabinet with dimensions 98x30x48 in. Optics are arranged to provide versatility in the observation and recording of boundaries by five optical techniques.

8—STRAIN INDICATOR: A new model M portable SR-4 strain indicator is announced by *Baldwin-Lima-Hamilton Corp.* The instrument is designed for operation with either batteries or ac power pack, which is optional equipment.

9—TESTING UNIT: Unit capable of extreme temperature ranges within a relatively short time has been developed by *Webber Mfg. Co., Inc.* Temperatures range on the standard unit from −80°F to +185°F. The dimensions of the chamber are 12x 12x12 in. Overall dimensions are 50 in. long, 26 in. high, and 20 in. deep.

10—RADIATION DETECTOR: It is possible in a single instrument to measure from ordinary background radiation to over 1500 R per hr. *Jordan Electronic Mfg. Co., Inc.*

11—FLO-METER: *Waukee Engineering Co.,* has announced a built-in-control valve version of the standard Waukee flow-meter. These models are available in 11 different sizes. Meters are available for

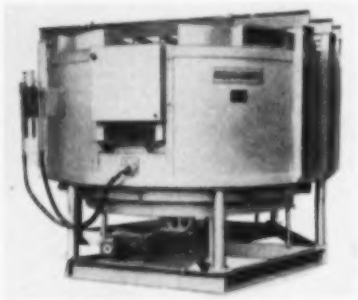


measuring air, ammonia, butane, carrier gas, hydrogen, oxygen, and others. Valves are located at top of the meter for easy reading.

12—GALVANOMETER: A redesigned portable galvanometer has been announced by *General Electric Co.* High sensitivity of the new equipment is made possible by alnico magnets which give a field of more than twice the strength obtainable with chrome-steel magnets previously used. The scale is marked 50-0-50 and 0-100 in 1 mm divisions.

13—AL SOLDERING: A new fluxing compound for use in soldering aluminum has been announced by *Essex Wire Corp.* It makes possible the noncorrosive soldering of aluminum alloys, except a few containing high percentages of silicon. Aluminum can be joined to other metals. It may be used for manual, dip or mechanical production soldering operations, either in powdered form as delivered or in molten form with equally satisfactory results.

14—ELECTRIC FURNACE: Designed to operate continually at temperatures to 2500°F, this furnace has a rating of 260 kw with a capacity of 1500 lb per hr. This electric rotary hearth furnace has been developed by *Hevi Duty Electric Co.* Silicon carbide rod type heating elements are arranged vertically and can be easily replaced while the furnace is hot.



15—CARBOY: Carboy tilter with attachment for chair hoist use for safer, faster, and easier pouring of acids from any size carboy or metal container is available from *General Scientific Equipment Co.* The safety air vent pouring spout assures a smooth flow with no spurts or splashes. It has a flow capacity of 5 gal per min.

16—STRAIN GAGE: The smallest SR-4 bonded resistance wire strain gage developed has been added to the various sizes and types made by *Baldwin-Lima-Hamilton Corp.* It has a gage length of 1/32 in. Nominal resistance is 120 ohms and the average gage factor is 1.4.

17—CAPACITROL: A new indicating controller featuring anticipating action has been added to the Wheelco model 200 series Capacitrol line. *Barber-Colman Co.*

18—MILLIVOLTMETER: A single point, double range indicating millivoltmeter pyrometer has been developed by *Minneapolis-Honeywell Regulator Co.* It incorporates a newly designed plug in galvanometer.

19—AQUAMETER: A high speed electronic device which titrates for moisture automatically, is available from *Beckman Instruments, Inc.* The instrument utilizes the Karl Fischer dead stop reaction for water measurement.

Free Literature

20—ROTOR MOTORS: Line of wound rotor motors, ½ to 1000 hp is covered in bulletin issued by *Louis Allis Co.* Performance curves and characteristics show how these units are suited to particular jobs in industry.

21—RUBBERIZED ABRASIVES: Burring, smoothing, and polishing operations of rubberized abrasives are described in catalog 53 available from *Cratex Mfg. Co.*

22—HIGH SPEED FURNACE: Bulletin SC-162 points out the advantages of high thermal head heating with gas or oil, for press forging, upsetting, extruding, stress relieving of ferrous and nonferrous metals. Basic furnace designs and burner arrangements are described in conjunction with the demands of overall or selective heating for semiautomatic or fully automatic production. *Surface Combustion Corp.*

23—IMMERSION HEATER: An electric immersion heater catalog that features a section of application data for calculating the power requirements for heating processing tanks has been prepared by the *Cleveland Process Co.* Two types of electric immersion heaters are fully described.

24—ZN DUST: Common applications of zinc dust are described in bulletin issued by Federated Metals div., *American Smelting & Refining Co.*

25—SOLDERING: Soldering irons and soldering iron tips for industrial applications are described in bulletin announced by *General Electric Co.* Photographs, diagrams, and tables describe soldering iron construction, application, ratings, and prices.

26—X-RAY ANALYSIS: Booklet entitled *Twenty Questions and Answers on X-ray Analysis* has been published by *North American Philips Co., Inc.*

27—INSTRUMENTATION: More than 300 technical case histories and general information articles on industrial instrumentation are listed in an alphabetical index published by *Minneapolis-Honeywell Regulator Co.*

28—TITANIUM: Properties of titanium and titanium alloys are contained in booklet issued by *Mallory-Sharon Titanium Corp.* Different production methods, general properties of various alloys, and specific qualities of five types of titanium and its alloys supplied by the company are several points covered.

29—HEAT TREATMENT: Annealing, hardening, martempering, austempering, and surface hardening are explained in detail in booklet published by *Meehanite Metal Corp.*

30—LABORATORY AIDS: Scientific apparatus and laboratory chemicals available from *Burrell Corp.* are listed in bulletin 328.

31—ELECTROPOLISHING: Bulletin is offered providing information on the theory and practice of electrolytic polishing of metallurgical samples, together with description of Buehler Electro Polisher. *Buehler Ltd.*

32—FINISHING: *Pelron Corp.'s* services and products for the finishing industry are described in bulletin just released. Studies and recommendations regarding metal cleaning, phosphating, are included.

Journal of Metals
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July

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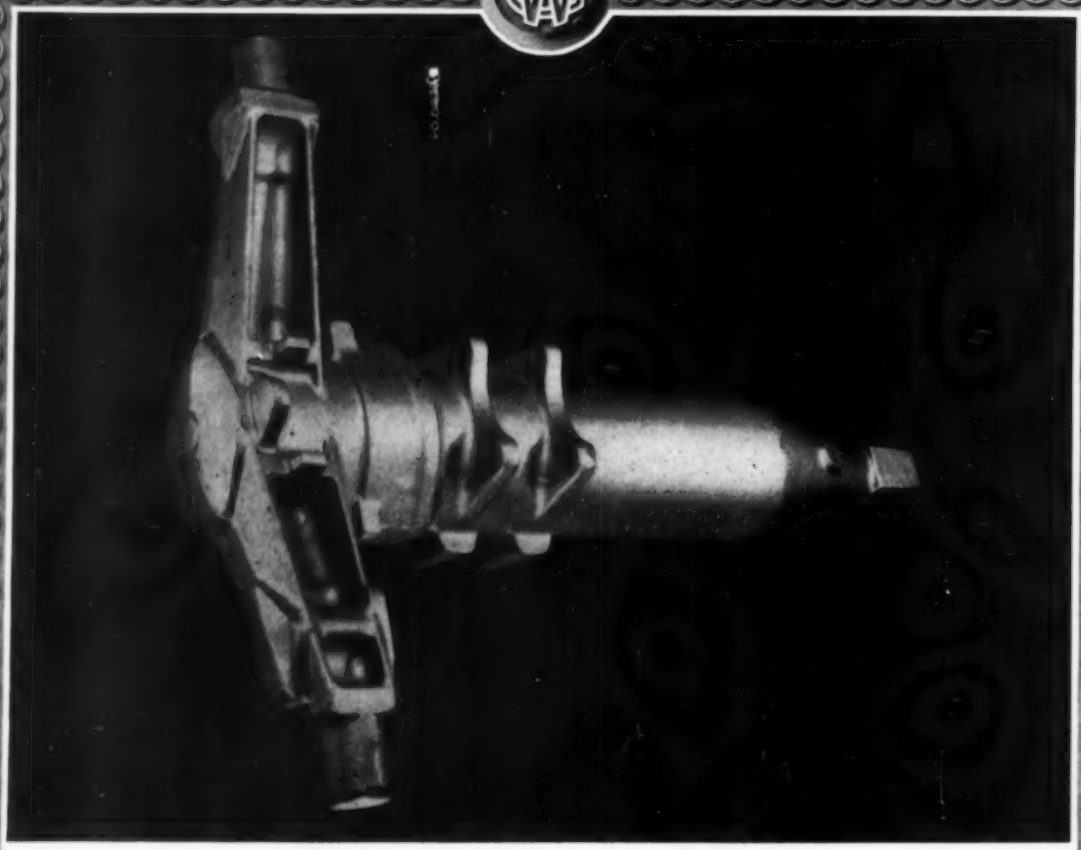

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Coming Events

July 5-10, International Institute of Welding, annual assembly, Technical University of Copenhagen, Copenhagen, Denmark.

July 21-28, International Union of Crystallography, general assembly and international congress, Paris.

Aug. 17-19, Society of Automotive Engineers, international west coast meeting, Vancouver, B. C.

Aug. 19-21, Western Electronic Show and Convention, San Francisco.

Sept. 11, AIME, St. Louis Local Section, Lead Belt Meeting, tour St. Joseph mines and mills.

Sep. 13-16, Electrochemical Chemical Society, Inc., Ocean Terrace Hotel, Wrightsville Beach, N. C.

Sept. 14-17, Society of Automotive Engineers, tractor and production forum, Milwaukee.

Sept. 16-18, Porcelain Enamel Institute, annual shop practice forum, Columbus.

Sept. 17-18, National Foundry Assn., annual meeting, New York.

Sept. 19, AIME, National Open Hearth Steel Committee, Chicago Section, golf party.

Sept. 21, ASME, Industrial Instruments and Regulators Div., and Instrument Society of America, exhibit and joint conference, Sherman Hotel, Chicago.

Sept. 21-22, Steel Founders' Society, fall meeting, Hot Springs, Va.

Sept. 21-23, American Mining Congress, Metal and Nonmetallic Mineral Mining Convention, Olympic Hotel, Seattle.

Sept. 28-30, National Electronic Conference, Chicago.

Sept. 28-30, ASME, Petroleum Mechanical Engineering Conference, Rice Hotel, Houston.

Oct. 5-7, ASME, fall meeting, Hotel Sheraton, Rochester.

Oct. 6, AIME, National Open Hearth Steel Committee, Buffalo Section.

Oct. 7, AIME, National Open Hearth Steel Committee, Chicago Section, Phil Smidt's.

Oct. 7-9, National Assn. of Consulting Engineers, south central region, Mayo Hotel, Tulsa, Okla.

Oct. 8-9, National Conference on Industrial Hydraulics, annual meeting, Hotel Sheraton, Chicago.

Oct. 9, AIME, St. Louis Local Section, St. Louis University, St. Louis, Mo.

Oct. 9, AIME, National Open Hearth Steel Committee, Eastern Section, fall meeting, Warwick Hotel, Philadelphia.

Oct. 15-17, AIME, National Open Hearth Steel Committee, Southwestern Section, fall meeting, Baker Hotel, Dallas, Texas.

Oct. 19-21, AIME, Institute of Metals Div., fall meeting, Hotel Allerton, Cleveland.

Oct. 19-23, National Metal Congress and Exhibition, Cleveland.

Oct. 22, AIME, St. Louis Local Section, Engineers Club of St. Louis and ASCE, Engineers Club, St. Louis.

Oct. 27, Assn. of Consulting Chemists & Chemical Engineers, 25th anniversary annual meeting, Hotel Belmont Plaza, New York.

Oct. 28-31, AIME, El Paso Fall meeting, El Paso.

Oct. 29-30, AIME Coal Div., ASME Fuels Div., Conrad Hilton Hotel, Chicago.

Oct. 29-31, National Council of State Board of Engineering Examiners, Annual Meeting, Plaza Hotel, San Antonio, Texas.

Oct. 30-31, AIME, National Open Hearth Steel Committee, Deshler-Wallick, Columbus.

Nov. 4-6, National Time and Nation Study and Management Clinic, Sheraton Hotel, Chicago.

Nov. 6, AIME, National Open Hearth Steel Committee, Pittsburgh Section, William Penn Hotel, Pittsburgh.

Nov. 13, AIME, St. Louis Section, coal meeting, York Hotel, St. Louis.

Nov. 29-Dec. 4, ASME, annual meeting, Statler Hotel, New York.

Dec. 2-4, AIME, Electric Furnace Steel Conference, Netherland-Plaza Hotel, Cincinnati.

Dec. 11, AIME, St. Louis Section, York Hotel, St. Louis.

Dec. 13-16, American Institute of Chemical Engineers, annual meeting, Hotel Jefferson, St. Louis.

Dec. 28-29, Annual Chemical Engineering Symposium, University of Michigan, Ann Arbor.

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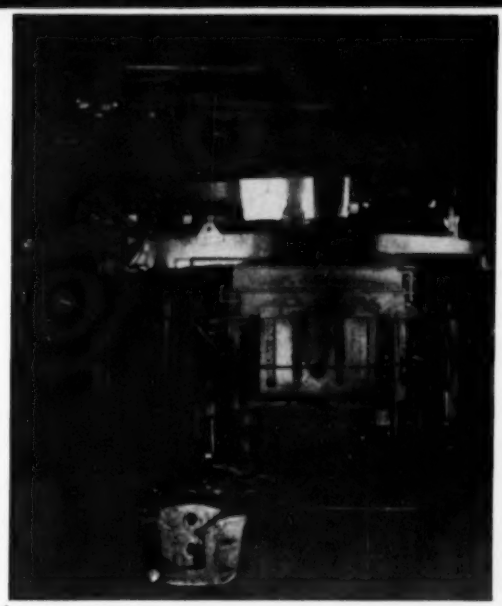

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Box F-17 JOURNAL OF METALS

Excellent openings in large modern research laboratory for (a) Metallurgical Engineer interested in laboratory development of foundry processes, particularly gray iron. Previous foundry experience helpful but not necessary. Main requisite is interest in foundry work. (b) Physical Metallurgist: Three to five years' experience with ferrous and nonferrous alloys. A broad opportunity to study current problems and to do development work on new processes. Both are permanent salaried positions.

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Box F-19 JOURNAL OF METALS

— Personnel Service —

THE following employment items are made available to AIME members on a nonprofit basis by the Engineering Societies Personnel Service, Inc., operating in cooperation with the Four Founder Societies. Local offices of the Personnel Service are at 8 W. 40th St., **New York 18;** 100 Farnsworth Ave., **Detroit;** 57 Post St., **San Francisco;** 84 E. Randolph St., **Chicago 1.** Applicants should address all mail to the proper key numbers in care of the New York office and include 6c in stamps for forwarding and returning application. The applicant agrees, if placed in a position by means of the Service, to pay the placement fee listed by the Service. AIME members may secure a weekly bulletin of positions available for $3.50 a quarter, $12 a year.

POSITIONS OPEN

Assistant to General Manager, 27 to 35, preferably metallurgist, or mechanical or chemical engineer with metallurgical experience, with experience in directing production of either a metal or plastic product, in either a foundry, machine shop or plastic injection molding plant. Knowledge of and experience in applying industrial engineering principles to above type of production. Company produces precision investment castings of industrial parts in ferrous and nonferrous metals. Duties include methods and quality improvements, plant layout and plant engineering, outside tool procurement, cost reduction, estimating and customer liaison. Salary $5400 to $7200 a year. Location, New York, N. Y. Y8740.

Metallurgical Engineer, graduate, experienced, to direct a broad research and development program related to the recovery and industrial application of strategic heavy metals. Strong background in chemistry with various experience in physical and hydrometallurgy are desirable. Should have a successful record in metallurgical research and be capable of formulating and directing a long-range program of basic importance to American industry. Salary open. Location, northern New York state. Y8708.

Assistant to President, not over 45, with metalworking experience in the stamping field covering sales, manufacturing, labor relations and other phases of management. Should possess qualifications required of a general manager. Salary open. Location, Detroit, Mich. Y8606-D8499.

Sales Engineer, 25 to 35, for a manufacturer of steel. Must have had at least two years' experience in sales contacts or steel application work. Will consider mill experience with sales potential. Knowledge of metallurgical processing on sales and application of alloy steel products such as ball bearings alloys desirable. Twenty per cent traveling. Car required. Salary, $5400 a year. Locations: Chicago, New York or Detroit. Employer will negotiate fee. R9846.

Metallurgist, recent graduate or better, for ferrous and nonferrous controls, process development and handling field complaints on tractors and Diesel engines. Salary up to $385 a month, depending on experience. Employer will negotiate fee. Location, western Chicago suburb. R9922.

Metallurgical Engineer, up to 40, for a manufacturer of automobiles. Must have had at least two years' experience in metallurgical laboratory or shop work. Duties will involve sheet metal welding and fabrication work in automotive body work. Salary up to $450 per month depending on experience. Location, Midwest. R9910.

Associate Metallurgist or Metallurgical Engineer, B.S. metallurgy, over 30, for fabrication of nonferrous components for nuclear reactors through known techniques or special techniques developed as needed. Must have had at least five years' experience use of vacuum techniques of melting and casting nonferrous alloys and small arc melting furnaces and induction furnaces. Knowledge of nonferrous metal fabrication. Atomic energy project. Salary $465 to $885 month. Location, Chicago. R9985.

EUROPE

Chemist, specializing in precious metals, also versed in mining, leaving in August on extensive European trip, no time limit, offers professional services.

Box F-20 JOURNAL OF METALS

QUALITY CONTROL SUPERVISOR

To take charge of Quality Control Department of a steel fabricating plant of 1,000 employees located in a northwestern Pennsylvania city of 25,000 people. He will be responsible for laboratory performance tests on material and products, proper care of instruments and gauges, maintaining inspection standards, reporting causes of defective material, and supervising departmental inspectors.

Applicants should be college graduates in engineering, science or metallurgy, or equivalent in practical experience. Machine shop, mechanical laboratory, and some drafting experience required. Knowledge of standard metals testing procedures and at least five years experience in inspection supervision for parts in process and finished products essential. Must be able to supervise inspectors and cooperate with production foremen and personnel. KNOWLEDGE OF STATISTICAL METHODS OF QUALITY CONTROL NOT REQUIRED.

The Company is a nationally known steel fabricating manufacturer producing essential equipment for public utilities, industries, transportation, and government agencies. Plant is expanding to provide additional facilities for manufacturing present products and those in the development stage.

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The same care and skill go into every ton of U·S·S CARILLOY steel that *you* buy, whether it's a giant alloy ingot or a few tons of special steel. Our experienced metallurgists keep a close check on *every* heat of steel to make sure it has the strength, hardness, toughness and machinability that's needed.

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Symposium on Fatigue with Emphasis on Statistical Approach-II. *American Society for Testing Materials.* $2.00, 91 pp., 1953.—Material presented here was originally given at the 55th annual meeting of the ASTM. Titles of the papers in the volume indicate that authors have given attention to the variation associated with their materials and to their measurements. Titles and authors are: Statistical Nature of the Fatigue Properties of SAE 4340 Steel Forgings, by J. T. Ransom and R. F. Mehl; Statistical Behavior of Fatigue Properties and the Influence of Metallurgical Factors, by E. Epremian and R. F. Mehl; A Statistical Interpretation of the Effect of Understressing on Fatigue Strength, by E. Epremian and R. F. Mehl; and Fatigue Properties of Large Specimens with Related Size and Statistical Effects, by O. J. Horger and H. R. Neifert.

Refractory Hard Metals, by Paul Schwarzkopf and Richard Kieffer, in collaboration with Werner Leszynski and Fritz Benesovsky. *MacMillan Co.* $10.00, 447 pp., 1953.—This book deals with the properties and preparation of the so-called hard metals: That is, the refractory and hard carbides, nitrides, borides, and silicides of transition metals. It also includes chapters devoted to the application of these substances as constituents of high temperature materials. The plan as finalized for this work involves publication of two books. This first is limited to coverage of structure, preparation, and properties: In other words, to a treatment of the substances which represent basic constituents of the metal-bonded products now widely used as tool and wear and heat resistant materials. Utilization of hard metals in materials for service in high temperatures is treated in this book. Chapters on this subject are considered by the authors to represent not only a critical evaluation of the usefulness of hard-metal compositions in applications such as turbine buckets or rocket nozzles, but to an extent also a general survey of the present status of development in the entire field of high temperature materials.

Metal Cleaning Bibliographical Abstracts, prepared by Jay C. Harris. *American Society For Testing Materials.* $4.25, 132 pp., 1953.—Material is presented in order to make published data on metal cleaning readily available to persons concerned with production, finishing, and maintenance of metal products. More than 1000 annotated references have been indexed. This new edition combines all references from the 1949 edition of Special Technical Publication 90 and its 1950 supplement. In addition several hundred new references bring the work up to date on all kinds of metal and most types of processes.

Tool Engineers Data Book, by Gerhard J. Gruen. *Reinhold Publishing Corp.* $5.50, 219 pp., 1953.—Access is offered to tables, formulas, constants, and specifications needed by the practicing tool engineer and designer. All explanations of theoretical concepts and other text matter have been omitted, as have obsolete material and tabular matter not directly related to the work of the tool engineer. Effort has been made to cover all latest developments with data on general properties of special alloys, heat treatment of special alloys and stainless steels, spot identification of metals, alloys and plastics, blank size diameters for shells and other information are included.

Engineering Manufacturing Methods, by Gilbert S. Schaller. *McGraw-Hill Book Co.* $7.00, 613 pp., 1953.—Engineering students will find a broad picture of the fundamentals of founding, machining, welding, hot and cold shaping, thermal treatment, and engineering materials. In the discussion of materials, some attention is given to light metals and plastics. Special emphasis is placed on recent technological developments in methods. In addition to literature reference a list of motion pictures and film strips is included.

Non-Ferrous Ore Dressing in the U. S. A. *The Organisation for European Economic Co-Operation,* 209 pp., 1953.—Material constitutes data in a report prepared by a Technical Assistance Mission of European experts. The group visited the U. S. under the auspices of the OEEC and the ECA. Object of the trip was to enable experts of various nationalities to study different aspects of the U. S. industry and make comparisons with practices in their own countries. The report is divided into two parts, i.e. a general analytical description of the ore dressing technique in the U. S., and a series of annexes giving the flowsheet and essential characteristics of the main plants visited by the mission.

Introduction to the Theory of Plasticity for Engineers, by Oscar Hoffman and George Sachs. *McGraw-Hill Book Co.* $6.50, 276 pp., 1953.—

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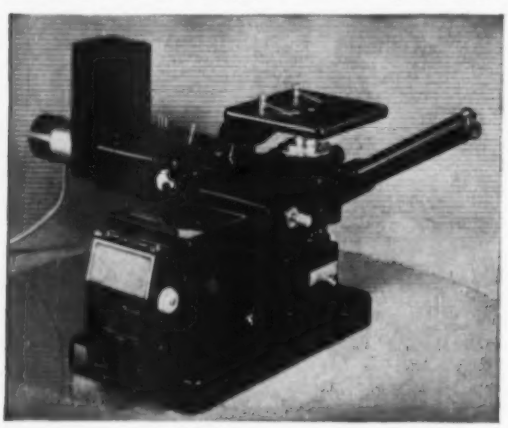

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Cast Bronze, by Harold J. Roast. *American Society for Metals.* $4.00, 458 pp., 1953.—The book represents 40 years of experience by the author and is aimed at the investor, executive, metallurgist, designing engineer, foundryman, salesman, purchaser, teacher, and historian. Patternmaking, coremaking, and molding are covered in respect to general principles. With exception of the chapters on architectural, beryllium, and nickel bronze, the author endeavored to keep within the field of his own experience.

Progress in Metal Physics, Vol. IV, edited by Bruce Chalmers. *Interscience Publishers, Inc.* $9.00, 403 pp., 1953.—Purpose of this series of books is to discuss and present authoritative information on the current state of knowledge in specialized aspects of the field that includes both physical metallurgy and metal physics. The intention is to cover over a period of several years all the more important aspects in which progress is being made. Historical background is discussed the first time a particular subject is introduced. Subsequent material on such subjects covers only more recent progress.

Introduction to Solid State Physics, by Charles Kittel. *John Wiley & Sons, Inc.* $7.00, 396 pp., 1953.—The text is intended as an introduction to solid state physics for seniors, and beginning graduate students in physics, chemistry, and engineering. In presentation the author assumes the reader has a general familiarity with modern atomic physics to the extent offered by undergraduate courses. While a course in quantum mechanics is not a prerequisite, the reader should have been exposed to the Planck radiations law, the de Broglie relation, the Bohr theory of the hydrogen atom, the Zeeman effect, and the wave equation for free particles.

High Temperature Alloys, by C. L. Clark. *Pitman Publishing Corp.* $7.50, 383 pp., 1953.—The author summarizes the present standards of knowledge of the behavior of alloys at elevated temperatures. Included is analysis and evaluation of literally thousands of articles written on the subject. Engineering aspects of alloy steel, rather than the theoretical, are emphasized. Considerable space is devoted to the most suitable procedures for selecting permissible working stresses and to the activities of the various specifications and code writing bodies. One chapter is devoted to the characteristics of the different types of high temperature failures. The book is intended as a text and reference work in view of plans of various colleges to establish courses dealing with the properties of alloys at elevated temperatures.

The European Steel Industry and the Wide-Strip Mill, prepared by the Industry div., Economic Commission for Europe. *The United Nations.* 100 pp., 1953.—This is the first of a series dealing with specialized problems affecting the European steel industry. It has been prepared by the Secretariat of the Economic div. for Europe with the advice and assistance of the Research and Planning div. The first chapter contains an account of past production and consumption trends in the U. S. and Western Europe. Future trends are analyzed. Industrial uses of wide strip, exports, and markets are also perused.

Metallurgical Engineering, Vol. I, by Reinhardt Schuhmann, Jr. *Addison-Wesley Press, Inc.* $7.50, 390 pp., 1953.—This book was developed from several years of teaching process metallurgy and metallurgical engineering courses at Massachusetts Institute of Technology. Organization stems from the idea that the most logical basis of classifying activities of metal extraction, and of classifying knowledge pertaining thereto, is according to the unit processes which are individual chemical steps used in extracting metals from their ores. Vol. I deals with engineering principles common to all unit processes. It includes extensive applications of the First Law of Thermodynamics which are presented to show the bridge between rigorous scientific statements of the principles and the frequently less rigorous engineering applications.

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Allis-Chalmers of Milwaukee is using Hevi Duty Controlled Atmosphere Furnaces to heat treat tools and dies made from high carbon, high chrome steels, 18-4-1, molybdenum, and cobalt high speed steels. Maintaining the exact surface carbon content of the tools and dies during heat treating is achieved with —

- A Hevi Duty Endothermic Atmosphere Generator supplying 500 cubic feet per hour of prepared atmosphere. With this controlled atmosphere, troublesome scale and decarburization or carburization of the surface is eliminated.
- A Hevi Duty Box Type Hardening Furnace, designed for temperatures to 2000° F., is used for preheating high speed steels and hardening carbon steels.
- A Hevi Duty High Temperature Furnace, designed for temperatures to 2600° F., is used to harden the high speed steels.

This combination assures you that tools and dies can be treated to exact hardness. Achieve better heat treating results by specifying Hevi Duty Furnaces. Write for Bulletin 153.

HEVI DUTY ELECTRIC COMPANY
MILWAUKEE 1, WISCONSIN
Heat Treating Furnaces... Electric Exclusively
Dry Type Transformers Constant Current Regulators

Industrial Notes . . .

• First mill designed for production rolling of uranium is in operation at Fernald, Ohio. The mill was designed and built by Birdsboro Steel Foundry & Machine Co., Birdsboro, Pa., for the Atomic Energy Commission. The mill is part of the AEC's new uranium production center at Fernald and is operated by the National Lead Co. of Ohio.

• **A new blast furnace is under construction at the American Steel & Wire Co., Cleveland div. of U. S. Steel Corp. Ground was broken for the blast furnace and special equipment which will take 14 months to build and integrate with the plant. Millions of gallons of water from the Cuyahoga River will be used to cool the furnace and wash the gas before it is reused. U. S. Steel is also planning to build a new foundry at a cost of $7.5 million at the site of one of the old ingot mold foundry units at its Edgar Thomson works in Braddock, Pa. Dismantling work on the old unit is reported to have started.**

• *All fabricating shops, machine shops, general offices and engineering departments of York Engineering & Construction Co. and York Gillespie Mfg. Co. have been relocated in one large plant at 39th St. and Allegheny Valley R.R., Pittsburgh. The new location accommodates all shop facilities under 10 and 15 ton cranes and has railroad spurs inside the building.*

• The physical metallurgy div. of the Melbourne branch of the Australian Institute of Metals will hold a symposium on the *Theory and Practice of Wire Drawing*, at Melbourne on Nov. 13, 1953. Several papers, including original work and reviews, have been contributed by Australian and English authors. They will be preprinted in booklet form and the November meeting devoted to discussion. Copies of preprints may be ordered from R. C. Gifkins, Bailieu Laboratory, University of Melbourne, Carlton, N. 3, Victoria, Australia. Charges are $1.70 post free by ordinary mail and $2.05 by airmail. Discussion on the papers is invited.

• George Sall Metals Co., Inc., opened its plant for the manufacture of nonferrous metal ingots from scrap materials on an eight-acre property at 2300 East Butler St., Philadelphia. Electric eye doors, latest materials handling equipment, air conditioning, ventilators, and communications equipment have been installed in the plant. Lead and zinc departments of the company are equipped with pots and kettles of 10,000 lb capacity each.

• **Full production is scheduled for International Minerals & Chemical Corp.'s new hydrochloric acid-magnesium oxide plant at Carlsbad, N. Mex., by the beginning of July. The chemical unit will produce large tonnages of hydrochloric acid and various high-purity magnesium oxide products from magnesium chloride brine, a byproduct resulting from production of potassium sulphate. Brine will be purified, concentrated and decomposed by heating to produce hydrochloric acid and magnesium oxide.**

• *Barium Steel Corp. contracted for a new type of ingot heat processing installation from the Selas Corp. of America that is scheduled for operation at Phoenix Iron & Steel Co., Phoenixville, Pa. by November 1953. Phoenix is a subsidiary of Barium. Cooperative tests by Barium and Selas indicate feasibility of various patent protected features and methods. The new method will reportedly reduce processing time to a fraction of conventional methods. It is also expected to reduce equipment and space requirements.*

• Two aluminum pipe lines totaling six miles were installed in the Permian Basin to be used for gathering sour gas. Cost was competitive with steel pipe. Installation employed the new automatic pipe welding system developed by Reynolds Metals Co., and permitted 40-ft lengths of 6-in. pipe to be lined up, welded, and lowered at an average of one every 3 min. In order to test the method's advantages, Reynolds is offering to work with oil companies on test pipe line installations in which Reynolds will furnish welding equipment and supervision.

• **Aluminum Ltd., parent company of Aluminum Co. Ltd., of Canada, announced contracts to supply a total of 600,000 tons of primary aluminum between 1953 to 1958 to Aluminum Co. of America and 186,000 tons during the same period to Kaiser Aluminum & Chemical Sales Inc. About 75 pct of the total is for delivery between 1955 and 1958. Authorization has been granted for immediate preparation of the ground site at Kitimat for two additional smelter potrooms, but actual construction is being deferred.**

• Defense Materials Procurement Agency has signed a contract for quick delivery with International Nickel Co. of Canada, Ltd. for 120 million lb of metallic nickel and 100 million lb of electrolytic copper. Deliveries will start in December 1953, at a rate of 2 million lb per month of nickel and 1,666,666 lb per month for copper. Completion of delivery is scheduled for 1958.

We'll build you a complete artillery shell forging plant and hand you the key

Salem-Brosius possesses a unique background of experience in the design and fabrication of shell forging plants which dates back well before World War II, and makes this organization the ideal source for such facilities. We are equipped to assume the entire contract, or any segment thereof. Salem-Brosius lays claim to being highly skilled in the design and construction of plants incorporating heating furnaces, descalers, preform presses, shell-forming mills, brooder furnaces, quench tanks, materials handling equipment, and all the hydraulic, pneumatic, electrical, and fuel systems and controls necessary to efficient and economical operation. If you are considering entry into the rapidly expanding business of producing ammunition to insure our country's safety, or contemplating expansion of your current operation, it will pay you to contact us. We'll build the complete plant and hand you the key or help you with any part of the problem. Write, wire or phone.

SALEM-BROSIUS, INC.

Sales and Executive Offices: 248 Fourth Avenue, Pittsburgh 22, Pa.
Brosius Division, Pittsburgh 15, Pa.
Salem Engineering Division, Salem, Ohio

This is a large, stationary, induction-heated high vacuum furnace of the type used for melting special alloys and certain pyroforic metals. Similar furnaces are used for sintering the new cermets such as titanium carbide with nickel, and aluminum oxide with chromium. In this type of furnace molten metal is cast—still in vacuum—through a plug in the bottom of the crucible actuated from outside through vacuum-sealed linkages. The furnace is a product of F. J. Stokes Machine Co., Philadelphia.

DURING May the Reports to the U. S. Atomic Energy Commission on Nuclear Power Reactor Technology were released by the AEC. The reports contained the results of four groups of companies making initial investigations into the feasibility of electric power generated by nuclear reactors. These four groups were: Commonwealth Edison Co., and Public Service Co. of Northern Illinois; Dow Chemical Co. and Detroit Edison Co.; Monsanto Chemical Co. and Union Electric Co.; Pacific Gas & Electric Co. and Bechtel Corp.

The AEC, from the time it took over the atomic energy program in 1947, has searched ways for industry to participate in development of nuclear fission. In 1950, Charles A. Thomas of Monsanto Chemical Co. proposed "that industry might with its own capital design, construct and operate nuclear reactors for production of plutonium and power." The AEC was interested.

Dow Chemical Co. made a similar proposal soon afterward. While considering the proposals, the Commission asked other concerns to offer suggestions. By the summer of 1951 the AEC had arranged for the four groups of two firms each to make at their own expense initial surveys of reactor technology.

The groups were to determine the engineering practicality of their designing, constructing, and operating dual-purpose reactors to produce fissionable material and power; examine the economic and technical aspects of building such reactors within the next few years; determine the research and development required; and submit recommendations as to industry's part in designing, building and operating such reactors.

The companies had available to them pertinent material on reactor technology. Study group personnel were allowed to consult with Commission and contractor scientists and engineers on classified and unclassified information. Twelve months were allotted to the studies. Of course, the reports contained restricted data. Restricted data condensed versions of the reports were distributed in October 1952. The group entered into the work with the knowledge that declassified versions of the work would probably be made public.

Because one of the objects of the study was to determine designs for reactors which might be constructed within the near future the companies for the most part were conservative in their concepts, stuck to "components of proved design, and conditions reasonably certain of early fulfillment."

The four groups were in agreement in the belief that dual purpose reactors are technically possible and could be operated in such a way that plutonium credit would reduce power cost. They also agreed that no reactor could be built in the near future which would be economic for power generation alone.

Commonwealth Edison Co. and Public Service Co. concluded that the heavy water reactor concept holds the best economic promise. However, important uncertainties in the shape of availability and cost of heavy water and practicality and cost of a chemical plant to process fuel elements enter into their thinking. A significant advantage may be had in the use of light water for moderator and coolant if sufficiently enriched uranium is available. Dependability of supply and cost of enriched fuel are uncertainties which may have to be resolved.

It is suggested that a gas-cooled reactor could provide a substantial start in atomic power with minimum interference with production reactors now under construction or in the planning stage. Fuel elements should be cheaper than some used in production reactors and they can be processed in existing chemical plants. Both reactor designs described in the report are considered feasible.

The Dow Chemical-Detroit Edison study reported that three changes were considered for conventional heavy water design. The first modification is a means for increasing the fuel and coolant temperatures of the reactor for practical power production by the use of liquid-metal coolants. Another change was development of a new type of metallurgical separations process. Another alteration was modification of the fuel system of the reactor to allow for closer integration with the separations process. The report states: These changes should be capable of incorporation in the heavy-water or graphite reactors, but the time and expense involved are considered to be as great as that required to develop the fast breeder

reactor with its low cost and virtually unlimited availability of fuel.

Monsanto Chemical Co. and Union Electric Co. began their investigations with the Hanford type reactor with the understanding that certain modifications would have to be made in order to convert the heat produced into commercially saleable power. Certain advantages were revealed for sodium as a high temperature coolant. Sodium-potassium eutectic, however, had the advantage over sodium of a lower melting point and thus a lower possible inlet temperature with a higher possible heat pickup.

Cost of reactor-produced electric power was found close to that from a modern coal-burning plant. It costs more to absorb heat by making steam acceptable for a turbogenerator than to dump the heat into a river. Also, the short five year amortization period assigned to reactor and power plants raises depreciation charges to a level, when combined with other cost increases, that offsets savings in fuel.

Pacific Gas & Electric Co. and Bechtel reported estimated costs for a 106.2-megawatt water-cooled thermal reactor at $41 million and for a 154.6-megawatt sodium-cooled fast reactor at $51 million. Total estimated yearly operating cost for the water-cooled reactor is figured at $1,580,000 and for the sodium-cooled reactor at $1,520,000.

North American Aviation, Inc., in the meanwhile, disclosed a design for a 8000 kw pilot plant to furnish information and engineering data. North American states that the pilot plant would take about two years to build and would furnish data to construct a 150,000 to 250,000 kw plant for about $300 per kw compared with $200 per kw for a coal plant.

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PITTSBURGH STEEL CO.'s steel producing capacity has jumped 48 pct by the method of enlarging the capacities of its 12 open hearth furnaces. Its steelmaking capacity is now fixed at 1,560,000 tons annually. The increased potential represents the largest percentage expansion for any company in recent years.

It cost Pittsburgh Steel some $9 million, or $16.31 per ton, to up production at the Monessen works where twelve, 150-ton per heat open hearths were raised to 250 tons each. Another phase of the expansion program was completed with the start of operations of six new soaking pits for heating steel ingots. A total of 15 furnaces is scheduled for installation at a cost of $3.5 million.

Following test runs, the firm's new hot rolled sheet mill will enter a break-in period before starting commercial operations. An installation for rolling cold rolled sheets is being constructed near the hot rolling mill and will go into production sometime later this year.

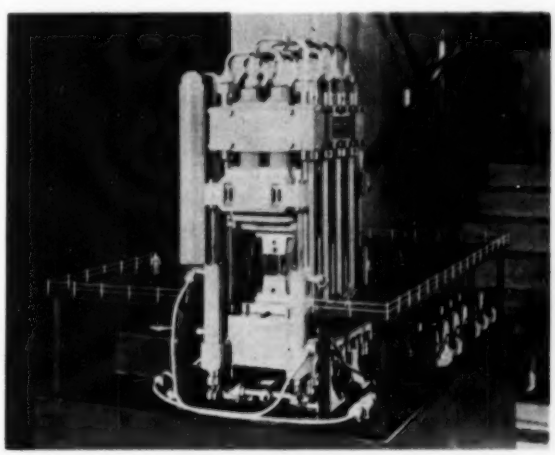
Pittsburgh's open hearths are equipped to make all grades of low, medium, high carbon and low alloy steel. In addition to enlarging the open hearths, three 300-ton ladle cranes were installed in the tapping pit. The charging floor was reinforced and the tapping pit floor was lowered three ft to handle 18 250-ton ladles.

BY 1975, so they say, the U. S. will need 45 pct more copper, 60 pct more lead, and 38 pct more zinc than in 1950. Consumption of these metals already has doubled since 1920. Battelle Memorial Institute, through its director, Clyde Williams, states that copper, lead, and zinc producers will meet increased demands although temporary shortages may occur from price difficulties or a national emergency. Battelle predicts a healthy long-range outlook for the metals based on the proved resourcefulness of copper, lead, and zinc producers in keeping pace with the expanding needs of the American economy.

"As in the past, copper, lead, and zinc producers will meet increased demands by making the most of technological advances that enable them to produce the greatest quantity of their products at the cheapest cost. Existing supplies of metals will be stretched by more efficient mining and processing techniques and by continued emphasis on recovery of scrap and waste suppression. Fundamental to future achievement, of course, is an economic environment that encourages the working of lower grade ores and the exploration of new deposits."

Mr. Williams asserts that improved drilling, blasting, and hauling techniques play a major part in mine yield improvement. Statistics indicate that ore tonnage output per man-hour more than doubled between 1935 and 1945. The Battelle director emphasized the importance of lower grade deposits. "The country is now obtaining most of its copper from ores that contain about 1 pct of the metal, compared to 5 pct in 1890."

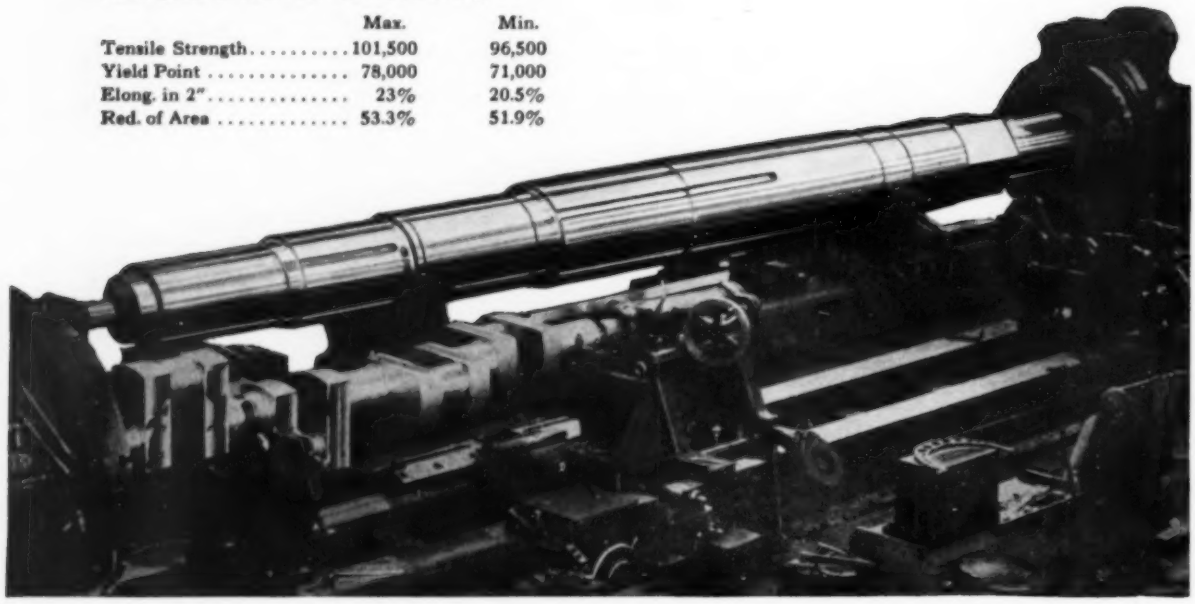
Engineering ingenuity made the mining of lower grade ores profitable and resulted in production from the huge deposits at Bisbee, Ariz., and Butte, Mont., Mr. Williams pointed out. "Such operations no doubt will continue as long as the price of copper remains sufficiently high to render them worth while."



The model represents a 35,000-ton forging press to be built by United Engineering & Foundry Co., Pittsburgh, for Aluminum Co. of America. The press will be housed at Alcoa's Cleveland works. It will be 52 ft from floor level and 21 ft below floor level. The press will have eight columns, 60 ft 8 in. overall and 34 in. diam.

NICKEL STEEL FORGING . . . a 58,950 lb. hoist drum shaft, 24" in diameter at its widest portion and 42' 9" long, produced by ERIE FORGE & STEEL CORP. Inspection revealed very fine grain. After being normalized and drawn, actual tensile tests in the longitudinal direction showed the following:

	Max.	Min.
Tensile Strength	101,500	96,500
Yield Point	78,000	71,000
Elong. in 2"	23%	20.5%
Red. of Area	53.3%	51.9%



How Erie Forge Obtains Superior Properties in Large Forgings

To develop high tensile and elastic properties in large forgings, such as this giant shaft, by heat treatment is much more difficult than with smaller forgings.

For even though dimensions of a large piece may allow liquid quenching, section sizes involved ordinarily limit the cooling rates.

Experience shows that superior mechanical properties in large forgings depend largely on suitable alloy content . . .

Fundamentally, that is why the output of ERIE FORGE & STEEL CORPORATION of Erie, Pennsylvania, includes scores of large forgings produced from nickel alloyed steel.

Nickel, either alone or in combination with other alloying elements, exerts highly beneficial influences. Its strengthening effect on ferrite is independent of carbon content or heat treatment of the steel, while its effectiveness in reducing the rate and temperature of the upper transformation, induces better response to the necessarily milder heat treatments used.

Nickel alloy steels may help you obtain peak performance from vital parts of *your* products or equipment. Send us the details of your problems for our suggestions. Write us now.

At the present time, nickel is available for end uses in defense and defense supporting industries. The remainder of the supply is available for some civilian applications and governmental stockpiling.

SYMBOL OF INCO SERVICE

THE INTERNATIONAL NICKEL COMPANY, INC. 67 WALL STREET NEW YORK 5, N. Y.

The Pacific Coast's third blast furnace went into operation at Kaiser Steel Corp.'s Fontana, Calif., plant. Identical to the two furnaces previously placed in operation at Fontana, the new unit raises West Coast pig iron capacity by 50 pct. The installation completes the major portion of Kaiser Steel's current $65 million expansion program. The new furnace has a rated capacity of 438,000 per year, bringing total plant rated capacity to 1,314,000 tons annually.

Extensive drying facilities will be built by Rust Engineering Co., at Aluminum Co. of America's new Rockdale, Texas works. The equipment will provide dried lignite to heat three large steam boilers of the Sandow power plant which will serve the plant. The Rockdale plant will represent the first use of lignite for industrial power production on such a large scale.

First carload of taconite concentrate produced by U. S. Steel Corp.'s Minnesota operation at Mountain Iron was shipped to blast furnaces. One concentration unit is operating with two others nearing completion.

A line of giant-sized carbon-bonded silicon crucibles, reported to be the world's largest, is being produced by Electro Refractories & Abrasives Corp. with the aid of a new process. The firm formerly was able to produce crucibles holding up to 400 lb of aluminum but with the new units capacity has been increased to 1100 lb, and even higher for heavier metals.

Brazilian Government Trade Bureau announced that a steel mill with an initial production of 50,000 tons of finished products per year will be built at Vitoria, Brazil. The plant is reportedly to be financed by Brazilian investors and the German Koeckner group. Initial development will cost some $8 million. Iron ore from Itabira, Brazil will be used. Peak production capacity will be developed over a four-year period.

Aluminum Co. of America liquidated its wholly-owned subsidiary, Alcoa Mining Co. to simplify the parent company's corporate structure. The subsidiary's operations were transferred to the newly created Mining div. of Alcoa. There is no change in function or responsibility of the mining company's personnel.

Dominion Foundries & Steel Ltd., signed a contract with Brassert Oxygen Technique A. G. of Zurich for construction of the first furnace in North America using the Brassert process for making steel. The method is reported to be cheaper than conventional processes and utilizes a converter similar to the Thomas converter widely used in Europe. Oxygen is blown in at the top in the Brassert process.

Iron production began from the second of two blast furnaces at the new U. S. Steel Corp. Fairless works near Morrisville, Pa. The bringing in of the new furnace, named the Hazel in honor of Mrs. Benjamin F. Fairless, gives the Fairless works an annual capacity of 1.2 million tons of iron.

Drift of Things

MANY comments have been received concerning the lack of interest shown by young people toward the mineral industries, as reported in this column in May. The occasion for the remarks was the disheartening turnout of mining and metallurgical aspirants among the high school students attending a New York guidance conference. That this is not solely a local condition was borne out several weeks ago at an MIED panel meeting held during the Pacific Northwest Metals and Minerals Conference in Seattle. There the discussion on *Fundamental Science in Metallurgical Education* led to the major problem — the vanishing enrollment in Mineral Science courses. According to Joseph Newton, professor of metallurgy at the University of Idaho, who reported this session for JOURNAL OF METALS, there was a feeling that discussion of courses and curricula was pointless when all statistics indicate that within a few years there will be no students to take the courses!

F. R. Morral, Kaiser Aluminum & Chemical Corp., who also took part in the MIED panel meeting, came across only one boy interested in metallurgy and none in mining, out of a group of 400 high school juniors in Spokane. He feels that high school age is perhaps not the best time to attempt to reach students, but that 5th to 8th graders are the most inquisitive. He also believes that public relations and information about science and engineering is as important to parents as to the young.

F. H. Rhodes, director of the School of Chemical and Metallurgical Engineering at Cornell University, agrees that it is unfortunate that so few capable young men are considering metallurgical engineering as a career. The shortage that exists, and indications are that it will get worse, poses a serious problem and impedes the expansion and improvement of metallurgical products needed to keep pace with the needs of the country and development of the industry. Quoting at random from his letter:

"My own feeling is that this apparent lack of interest is not so much lack of interest as lack of information. Most of the guidance counselors in our high schools and preparatory schools have not had any training in engineering nor any very great contact with engineering as a profession. . . Very often a young man who has shown real aptitude in mathematics and science is properly advised by his guidance counselor to consider some field of engineering as a career . . . the suggested choice is usually limited to mechanical, civil, electrical or chemical engineering. . .

"There has been no such public recognition of metallurgical engineering and no very great publicity as to the recent and impending marked advances in that field and the opportunities there. . .

"I do not see very clearly how the AIME as an organization can do a great deal to remedy this situation, but I do believe that individual members and associates of your society can do a great deal to help as individuals and as companies. Any effective effort to publicize metallurgical engineering as a profession should be aimed at young men in about the second year in preparatory school and perhaps to the parents of these young men as well as to the schools themselves. . .

"I think another way in which an effective effort can be made is through the advertising of the several companies engaged in the metallurgical field. . . I see no reason why a well designed and well presented advertisement, emphasizing the possibilities of a career in metallurgy, should not be just as effective as institutional advertising as are many of the advertising programs now in effect."

Professor Philip B. Bucky of Columbia University believes the subject might well be seriously discussed by representatives of industry and the teaching profession at the next AIME meeting.

All agree that the situation is serious. There is even agreement as to methods of educating the masses as to the existence, opportunity, and yes, even the glamor, of mining and metallurgical engineering. It would take time and money to carry out a program — but the results should far overshadow the investment.

LAST month we attended a showing of Electro Metallurgical Co.'s (UC&C) new Marietta, Ohio plant, and joined the ranks of the few ever permitted to tour a ferroalloy plant. Many are the stories told of those who vainly attempted the feat. At any rate, after the tour, we found ourselves seated in a large lecture room, listening to Dr. A. B. Kinzel, vice-president, ElectroMet, speak on *Research, Development, and Use of the New Alloys and Metals.* In discussing the new extra low carbon ferrochrome now being marketed, he mentioned the research and development that was necessary for reducing the carbon content from three parts in 10,000 to one part in 10,000. Our mind wandered back to the IMD Lecture in 1950, when Earle Schumacher discussed *Metallurgy Behind the Decimal Point.* And it was not so many years ago that metallurgists were busily and successfully engaged in adding "9's" to the purity of zinc.

Often the cry is for the new and totally different, yet so much can still be done with what is at hand. Whatever the goal, it is becoming more universally realized that research is the key to future technology. Of course, the scientist and research engineer have been aware of this, but production people and management have tended to accept the idea slowly. Perhaps it is the impatience of the practical operator to get something working, perhaps the aloof attitude of the scientist, that prevented mutual understanding. However, with many large metallurgical industries taking the lead in giving research its earned place, all industry must soon realize the significance. Perhaps some day soon a research team will come up with a report on *What Does a Metallurgist Do* that we can use to answer the question most frequently asked of us. Our stock answer to date is that a metallurgist can do anything he is called upon to do. Well, almost anything.

Alvin S. Cohan

Trends In Refractory Practice —

Monolithic Linings In Furnace Spouts

Presented at the National Open Hearth Steel Conference,
Buffalo, Apr. 20, 1953

Working Conditions Improved at Armco Steel Corp.

by V. W. Jones

MUD linings in furnace spouts proved unsatisfactory at Armco Steel Corp.'s Middletown, Ohio plant. Finally, after wrestling with the problem for several years, management took steps because the casting pit became congested when high hot metal practice started. A tabulation of involved factors illustrates the reasons for introducing improvements.

1—There were numerous minor injuries, several close majors, and eventually a major injury caused by the limited working space in the casting pit.

2—Large amount of clay that washed into the ladle did not contribute to quality.

3—The job of preparing the runner was disagreeable due to the heat and congestion in the spot in which the work was done.

4—The operation was expensive because of short life of brick linings.

First move toward a remedy was an attempt to correct the safety hazard and improve the working conditions by setting aside one area in the pit as a runner repair station. Reasoning was that by putting the runners and mud boxes in a spot away from hot thimbles, hot ladles and hot drags of molds, at least half of the problem could be corrected. This move was unsatisfactory because it added much work to the ladle cranes in transporting runners, making it impossible to keep up with the job.

Fig. 1—In practice a lining of ladle brick covers the bottom of the runner and extends up the sides to the studs. Studs are rail spikes placed 2 in. apart, extending 8 in. from top flange in center section and 14 in. from flange at end sections.

Fig. 2—Basic lining material is rammed in the bottom of the runner, and the completed runner allowed to dry.

A trial began of various materials for runner linings to find something that would lend itself to easier and faster cleaning and preparing. A material was finally found and a practice arrived at that has greatly improved the condition from all four standpoints. Work on the present practice was started in No. 1 open hearth shop but reached its present stage of development in the No. 2 open hearth shop. Both shops now use the method.

The basic lined runner expense was 62½ pct of the brick lined runner expense at the time of the

V. W. JONES is Superintendent of Steel Plant Production at the Middletown, Ohio works of Armco Steel Corp.

Fig. 3—Completed runner with form burned out is ready to use. Runners that have been in service are patched, and a thin slurry of loam added.

changeover in No. 2 open hearth shop. The cost of materials and labor in building new runners was the only item considered—no credit is allowed for decrease in the work of furnace helpers. The practice has improved considerably since the time of the cost comparison so that it is reasonable to say that the present cost is less than half that of the old method.

Runners have been in service with more than 500 heats without a relining job. The total maintenance cost of a runner with 500 heats has not yet reached the cost of the original lining, thus the point at which it will be economical to tear out and reline is unknown. There has been considerably less cutting of runner ends when lined with this material so there has been a decrease in casting cost. The time spent in cleaning and repairing runners by second helpers has been more than cut in half. Helpers appreciate the decrease in the disagreeable work of their job and improvement in safety conditions.

Monolithic Linings Successful at Crucible Steel Co.

by G. M. Burrier

MONOLITHIC linings were first used at Midland works in furnace spouts, short spouts, and ladle spouts in 1949. The practice came about as a result of the damage done to the hearth beams of the old open hearth furnaces in previous years.

For want of a better idea the first short spouts were built of reclaimed brick and required continual maintenance. The design of the first monolithic short spout came through the company's suggestion plan and was drawn up by the bricklayer with the unpleasant task of maintaining the short spout. Success of the monolithic lining caused it to spread rapidly to the furnace spouts, front flush castings, ladle slag spouts, hot metal spouts, etc.

During the past three years the only equipment reverted to brick linings was the hot metal spout where the problem was kish and metal buildup and burning of the front casting rather than lining wear.

Fig. 1 shows this short spout on a newly constructed furnace.

One-in. plate is welded to the furnace casting to frame the monolithic material. Five small lugs are welded on each side of the spout to hold wooden forms used for installation. The spout is cast in two sections with the top half overhanging the bottom by 6 in. This is done so that any damage done by excessively long drawing time will damage only half of the spout. The lower half of the spout covers the hearth beam and is good for 500 to 1000 heats

G. M. BURRIER is Assistant General Manager of Crucible Steel Co. of America's Midland works.

Fig. 1—Lower half of this short spout shown on a newly constructed furnace is good for 500 to 1000 heats without maintenance. Top half of spout usually is replaced after 200 heats.

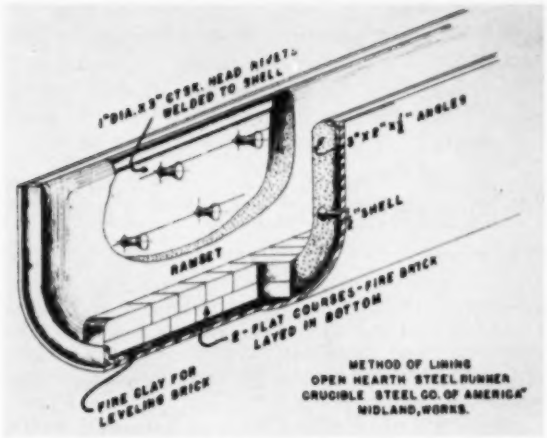

1"DIA. X 3" CTSK. HEAD RIVET - WELDED TO SHELL
3"X3"X½" ANGLES
SHELL
RAMMET
8 - FLAT COURSES - FIRE BRICK LAYED IN BOTTOM
FIRE CLAY FOR LEVELING BRICK
METHOD OF LINING OPEN HEARTH STEEL RUNNER CRUCIBLE STEEL CO. OF AMERICA" MIDLAND, WORKS.

Fig. 2—Cutaway view of a monolithic furnace spout shows the method of lining and construction detail. The spouts are 2 ft high and 14 ft long.

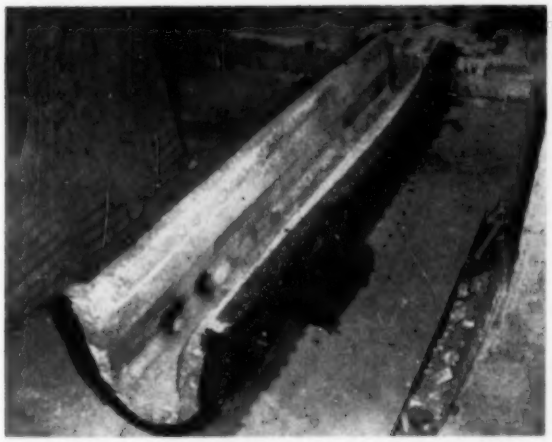


Fig. 3—This newly rammed spout is ready for service. Length of the runner is slurried with fire clay between heats.

without maintenance. The top half requires about one superficial repair per week plus a repair after each period of lengthy draining time. This type repair takes about 15 min after material is ready. The top half is usually completely replaced after each roof or approximately 200 heats. The pipe and hose in Fig. 1 were steam lines used to heat the rammed furnace hearth since the furnace was not put into operation immediately after completion.

Fig. 2 shows a cutaway view of a monolithic furnace spout. The spouts are 2 ft high, made of ½-in. plate with a curved bottom. Wide rear platforms cause the furnace spout to be 14 ft long. Both ends of the spout have 3 ft x 3 ft x ½ in. angles. The sides are topped by 3 ft x 2½ ft x ½ in. angles. The spout has one row of studs 6 in. below the top with 12 in. spacing. The second row is 10 in. down with 12 in. spacing and staggered with the first row.

In lining, the bottom of the spout is leveled with fire clay. One flat course of 9-in. clay brick is laid. Then a second course of 13x6x3 ft are laid. Precut wooden forms are inserted. Thirty sacks of magnesia type material for bottoms are mixed with six sacks of cement. During the past two years mixes sufficiently wet to cast and dry enough for bottom installation have been used. There is a slight advantage to keeping the mix dry.

The practice arrived at has been to make the mix slightly wetter than the bottom mix and to ram in manually. The monolithic lining is 4 in. thick at the top and runs into the 6-in. thick bottom with a side thickness of 5 in. The lining is installed by a brick layer and a helper.

Because of the slow turnover of spouts, several days or possibly weeks of air drying will follow. One spare spout is kept in the shop fully rammed for any emergency. Spout lining life is quoted at 200 heats but often runs greatly in excess of this figure. The entire length of the runner is slurried with fire clay between heats.

Fig. 3 shows a newly rammed spout. One of the main advantages of the monolithic spout is the ease with which scrap can be removed after each heat. With the furnace spout-short spout combination leaky joints have dropped from the *occasional* level to the *almost never* level. The savings in bricklaying man hours and the resultant effect of no damage to equipment have been considerable. Another advantage that is hard to evaluate is that this phase of the operation, once a daily problem of supervision, now runs by itself with little supervisory attention.

Practice Adopted at Bethlehem Steel Co.

by J. C. MacNeill

FIRST use of a monolithic refractory-lined spout at Bethlehem Steel Co., Bethlehem, Pa., was made in October 1951, when the plant installed one spout in No. 4 open hearth. Results obtained on this installation led to 100 pct use in No. 2 and No. 4 open hearths. The advantages of the monolithic refractory over the brick-lined spouts are longer life, less trouble removing skull, and quicker lining and drying, enabling the second helper to finish his work faster.

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Spouts are prepared in the following manner. Three rows of studs, 4x½ in. on 6-in. centers are

Fig. 1—After studs are welded on spout, refractory is rammed in place to a depth of 4 in. on sides and 8 in. on bottom. Average spout life has been 259 heats.

welded on the main body of the spout. The spout ends are studded completely, using 5-in. centers. Studding is done with a stud gun connected to regular welding units.

The refractory is placed in the spout and rammed in place on bottom and sides with a chisel-type tool, to the depth of 4 in. on sides and 8 in. on bottom. Ramming must be as tight as possible to eliminate voids. Bottom thickness is measured by inserting a thin rod through the material, marking it, and measuring with a rule. Vent holes are made with a tenpenny nail and three holes are burnt in the bottom of the main spout to allow water or steam to escape while the spout is being dried.

Fig. 2—This finished spout will be lined with a mud slurry of Phillipsburg loam. Spout life depends on proper drying, and the care given by furnace crews.

Table I. Cost Comparison Between Monolithic Refractory and Brick

	Monolithic Refractory	Brick Lined
Material	$210.00	$28.13
Studs	23.10	—
Labor Studding	10.50	lining 14.00
Ramming	21.00	
Total	$264.60	42.13
Cost per Heat	1.02	0.94
Difference	±0.08	

Spouts are dried with coke oven gas, using a pipe burner placed along each side of the spout. It has been found better to dry spouts from the outside of shell. The moisture is driven out through the vent holes and is not trapped inside. It is also an insurance for safe drying throughout the whole monolithic refractory. According to experience it pays to have the material dried and set before using. Three days are required to dry out a spout. By using a drying oven or more burners on the outside the drying time can be cut down.

After the spouts are ready for use, they are lined in the conventional manner, using a mud slurry of Phillipsburg loam. Placed on the lip end overhanging the ladle are two 1-in. clay bricks covered with loam. This helps to keep the steel from cutting back the lip. To date only two of the ladle end lips have required replacement.

Life of these spouts varies considerably, ranging from 106 to 548 heats, with an average to date of 259 heats. Average life on brick spouts has been 45 heats. Life of these monolithic spouts depends on drying and the care given by the furnace crews. Spouts with the shortest life were found to be on *underfired* or *not properly cleaned after each heat.*

When one of these spouts is dug out for relining, it is necessary to replace only 20 pct of the studs. Small replacement is attributed to spout wearing through in different areas and to steel adhered to studs being burnt off. Patching over these thin sections was tried but proved unsuccessful. The new material does not adhere to the old and washes off after several heats. Experiments are continuing with methods to patch these worn areas so that it won't be necessary to reline the entire spout.

Advantages of a monolithic refractory spout over the conventional brick-lined type are easier removal of skull, easier lining, quicker drying, and less crane handling. Since this type of lining was adopted there has been no trouble in keeping an extra spout on hand. With brick-lined spouts it was impossible to keep an extra spout because someone was always using it when steel stuck in the brick joints and there was trouble cleaning it.

Difference in cost per heat is more than made up by the releasing of cranes for other work. A comparison of costs between monolithic and brick-lined refractory is shown in Table I.

A longer average life will be obtained by patching bad spots in the runner. All material has been removed to date when a bad spot develops. Now, it is unnecessary to remove all material, because digging out around a bad spot and undercutting the old material will make it possible to patch successfully.

Open Hearth Slag Removed Economically By

B-L-A-S-T-I-N-G

by J. O. Dague

THE Lackawanna, N. Y., plant of Bethlehem Steel Co. uses explosives exclusively for open hearth slag removal. Before standardizing on blasting slag, various methods were tried.

Years ago, slag was laboriously chipped out with sledge hammer and bar. With the introduction of compressed-air tools, concrete busters were substituted. Both methods were expensive and time consuming. About 12 years ago, Bethlehem experimented with slag removal by blasting with carbon dioxide bombs. This method necessitated cooling the slag and drilling a vertical bore hole from the top of the slag. This was unsatisfactory because of excessive cooling time and the action of the blast. The relatively slow, heaving action of the carbon dioxide bombs only broke the slag into large pieces. It was often necessary to redrill and shoot the larger pieces.

During this period, experiments were tried with *cast-in* bore holes. Hollow tile were placed in the slag pockets at rebuild; they collapsed. Holes were also formed out of different refractories; they either collapsed or the slag leaked into the formed space.

The carbon dioxide bomb blasting practice was followed by the practice of *pulling* the slag, using wire rope. To accomplish this, conventional sand walls were constructed parallel to, but separated from the slag pocket walls. The area between was filled with sand. A heavy layer of sand was placed on the bottom of the pocket and also on the sloping breast leading to the checkers.

Theoretically, the sand walls confined the slag. When the furnace was down for rebuild, the sand was removed from the sides and part of the bottom; 1⅛ or 1¼-in. wire rope was wrapped around the slag and the entire unit pulled by pit crane. The slag was loaded on special heavy railway cars and removed to the slag dump. There it usually had to be broken for final disposal.

J. O. DAGUE is Assistant Mechanical Superintendent of Bethlehem Steel Co.'s Lackawanna, N. Y. plant. This paper was presented at the AIME National Open Hearth Steel Conference, Buffalo, Apr. 20, 1953.

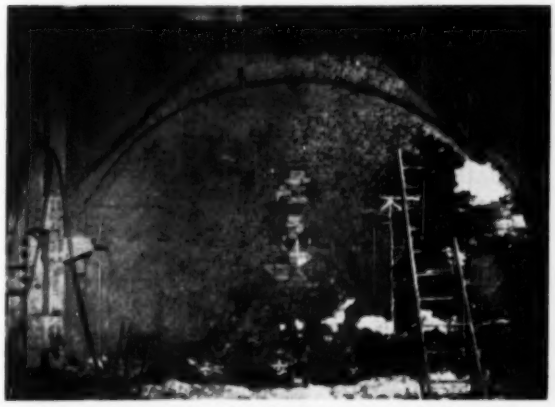

Fig. 1—First step in slag blasting is removal of a few bricks from slag pocket bulkhead to measure the height of the slag.

In practice, the slag often went through the sand walls and bonded into the slag pocket walls. Under the best conditions, higher slags were likely to break. This required additional digging and another pulling.

To speed this operation, rails were *cast* into the slag pocket during rebuild, with the thought that the slag would fuse around the rails. It was hoped that by lifting on the protruding rail ends it would be possible to pull the slag. After a furnace campaign, all that remained of the rails were the ends extending beyond the bulkhead. The necessary special shifting of railway cars, and the pit crane service required in the *pulling* method, caused delay either to the rebuild or to pit operations.

With the introduction of trucks and bulldozers to the open hearth pit, the previous methods of slag removal became incongruous. There were two separate systems to handle the same basic problem.

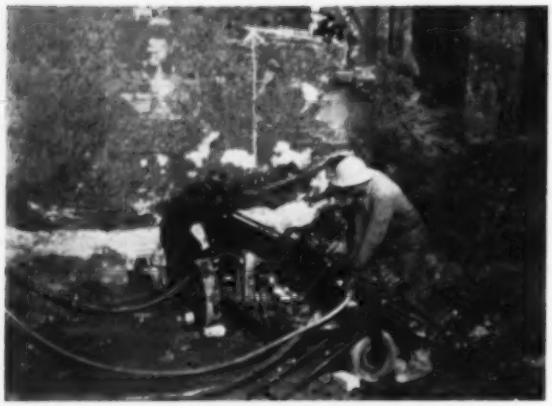

Fig. 2—Holes are drilled horizontally into the slag using an air-operated wagon drill and hollow drill rod. Cooling water is passed through the drill rod.

Fig. 3—Lightweight flexible cable is inserted into the holes to prevent plugging before blasting time by flaking slag. Water is fed into the tube overnight.

Crane and railway cars were used to remove the slag. Then, for efficiency, dozers and trucks were used to remove the teardown rubble.

The combination of dozer and truck was so vastly superior that it was natural to look for a method of breaking the slag into pieces small enough to handle by this method. In September 1949, experiments were started with horizontal drilling and blasting of slag.

The choice of drilling equipment was fortunate. *Wagon drills* were employed. No extensive change in drilling practice has taken place since the first attempt.

The first blasting try blew down the entire end of the furnace, arches, port roof and all. After that first fiasco, methods were standardized to suit conditions. In two years of slag removal exclusively by blasting, methods haven't changed appreciably.

Properties of the slag are attributed to its chemical analysis and mineral content, and to temperature of the slag pocket. It was found that pitch-fired furnaces lay down a slag that flakes on cooling and breaks rather easily. A furnace with basic ends lays down a hard, tough slag.

The practices as described are those that have been successful under conditions at Lackawanna. There have been no changes in furnace construction. It is still practice to build sand walls and sand the bottoms as when pulling the slag.

Procedures

The first steps in blasting the slag take place the day before the furnace comes down for rebuild. A few bricks are removed from the slag pocket bulkhead to measure the height of the slag. Fig. 1 presents a view of this operation.

Three 2½-in. holes are drilled horizontally into the slag. These holes are located so that two thirds of the slag is above the holes, one third below. Horizontally, one hole is in the center of the slag pocket. The other two, one on each side, are located one half the distance from the center hole to the inside edge of the sand wall. The center hole is drilled to 80 pct of the depth of the pocket; the side holes, 50 pct. Holes are drilled with a standard air-operated wagon drill. The drill rod is hollow, to permit the passage of cooling water. Fig. 2 shows the drilling arrangement.

Some difficulties were experienced with the slag flaking off and plugging the holes. A 2½-in. lightweight flexible conduit is now inserted to preserve the holes until blasting time. This conduit is similar to flexible electric conduit. However, it is cheaper and lighter. Insertion of one of these tubes is shown in Fig. 3. A trickle of water is continuously fed into the tube overnight.

After the furnace is tapped, the doors and frames are removed and the roof is dropped. Then slag pocket bulkheads are pushed in by a dozer. These operations require about two hr, during which the slag is cooling.

The third hour after tap, a substantial stream of water, giving a local cooling effect, is fed into the holes. The blaster sees the slag for the first time during this period. He checks height and condition of the slag and looks for any *hickeys* bonded into the walls plotting his charges accordingly.

On the average, the blaster charges 1½ lb per ft in the center hole. Half this, or ¾ lb per ft, is charged in the side holes. This amount of explosive equals ½ lb per cu yard.

The explosive used is an ammonium-nitrate, heat resistant, low sensitive blasting agent, requiring a special primer for detonation. It is a blasting agent originally developed for seismic prospecting. It is packed 1 lb to a 2½ in. diam can, and is approxi-

Fig. 4—Charges are made up complete before charging of holes, and the three caps wired in series. Charges are shown laid out before slag pocket.

mately 15 pct stronger than a 60 pct Red Cross Extra dynamite. The charge contains no nitroglycerine, is nonfreezing, and is not classed as an explosive.

One can of primer is used in each hole on the end of the charge nearest the open end. Detonating fuse is attached to the can of primer. This terminates in a conventional blasting cap.

The charges are made up complete before charging of holes. The three caps are wired in series. To gain time the wiring is tested and connected to the twist handle type blasting machine. For safety, the handle is carried in the blaster's pocket. Fig. 4 shows a layout of the charges in front of the slag pocket.

The holes are charged between 3 and 4 hr after tap. First the blaster blows the typical dynamiter's whistle. When all is clear, two men charge the outside holes simultaneously. One man then charges the center hole and runs into the clear. The third man then shoots the charges. In Fig. 5 the blaster just finished charging the center hole and is turning from the slag face to run into the clear.

The average time from start of charging to blast is 27 sec. Speed is the keynote, because of heat of bore holes. Therefore, no stemming is employed. No heat resistant protection is used for the primacord. From experience, the weakest link in the chain is the primer can. This primer, in an unstemmed hole, will take less prolonged heat than the primacord.

There has never been a premature blast. A few holes have failed because of the can of primer *gassing off* and failing to detonate.

Fig. 6 shows the slag a few moments after blasting. There are no large pieces. Bulldozers proceed immediately to load out the slag as in Fig. 7.

The average time, from start of furnace tap to slag removed, is 7 hr. Five to seven-ft slags are out in 6 hr. The 10 and 12-ft slags, as illustrated, take a little more than 8 hr.

Advantages and Disadvantages

There are many advantages to slag shooting. The most obvious is time. It took an average of 12 to 16 hr for slag removal under former methods. Very high slags often required 24 hr.

Slag blasting is cheaper. It requires less labor. Cheap truck transportation substitutes for expensive railway handling. Blasted slag is handled only once. Pieces are small enough to use directly as fill.

Fig. 5—The center hole has just been charged, and the blaster heads for the clear. Two men charge the outside holes simultaneously.

Fig. 6—This is the scene a few moments after blasting. Average time from start of charging, Fig. 5, to blast is 27 sec. There are no large pieces.

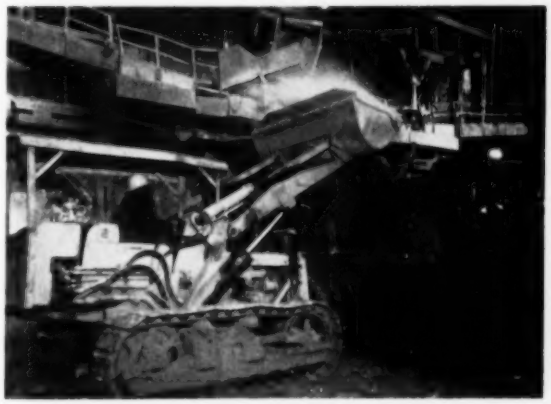

Fig. 7—Slag is loaded out immediately by bulldozers. Removal of blasted slag is hard on machinery, and rugged equipment is necessary.

Slag blasting saves brick. When pulling slag, it was found by bitter experience that salvaged brick could not be used in sand walls. Slag broke through salvaged brick sand walls and bonded into slag pocket walls. By blasting, all sand walls are now installed with salvaged brick.

Blasting helps tonnage. It requires no crane service and thus does not interfere with pit operations. By shooting, much higher slags are permissible. The slag is not pulled as often and thus permits more *quick roofs* in proportion to rebuilds. A furnace never has to be taken off for high slag.

There are a few disadvantages to blasting. A few extra brick are blown out occasionally and this requires a small amount of extra patching. However, a good arch has not been blown down since the first attempt. Removal of blasted slag is hard on machinery. It requires a very rugged machine.

At first there was concern about the possible shock damage to buildings. A seismograph was set up on a building column adjacent to a slag pocket, and under a rail joint. It was found that there was less shock caused by the blasting than that caused by a crane passing over the joint.

Advantages are savings in time and money, greater availability, and higher production; disadvantages are practically nil.

Vancouver Steel Co., Ltd., Uses Mullite Roofs on Electric Furnaces

by G. R. Heffernan

MULLITE deserves consideration from electric furnace operators where high furnace productivity is important and a large share of brick costs can be traced to freight rates. Vancouver Steel Co., Ltd., found mullite more than satisfactory for its operations after a protracted period of trial and error and disappointment. Fig. 1 shows a mullite roof being removed after 350 heats.

Vancouver Steel operates a 4000-kva, 11-ft diam Volta swing roof furnace. The diameter of the roof, inside the water cooled roof ring, is 9 ft 7 in. Voltage taps provided are 260, 200, 140, and 110 v. The furnace is equipped with 12-in. graphite electrodes. Electrode regulation is by means of contactor type equipment. Steel is made with a modified single slag process with 90 pct of the production in semi-killed ingots. Production is 3100 tons per month, approx.

The furnace is lined wih 13½-in. metal encased, chemically bonded magnesite-chrome bricks to the slag line. Below the slag line dead burned magnesite bricks were used but have been replaced with chemically bonded magnesite-chrome bricks with good results and a small cost saving. Bottoms are made of air-setting magnesite ramming mixes. A 1-yard cement mixer is used to provide a uniform mix and the bottoms are rammed into place with an air hammer.

In making bottom, a parting board is used down the center of the furnace and all the material for one half is shovelled in and rammed in place. The parting board is then removed; the leading edge of the new bottom is undercut at about a 45° angle, and the other half of the bottom is shovelled in and rammed. The bottom is dried from 8 to 12 hr under an oil torch and is brought up to approximately 1000°F. Electrode butts are then placed in a T formation on the bottom. Temperature is brought up at the rate of approximately 250°F per hr to 2900° to 3000°F where it is held for 8 hr. Care is taken not to let the temperature drop back once the ceramic bond is started. The first charge of selected scrap is carefully placed with the magnet so as not to break through the thin ceramic crust.

Roof Practice

The main departure at Vancouver Steel from what is recognized as standard electric furnace practice is in roof brick. Here 13½-in. silica special shapes had been used with normal results—an average of 112.4 heats over 33 roofs. Despite this average, which in most cases would be satisfactory, Vancouver found roof refractory costs unduly high, primarily because of high freight charges in transporting the silica brick from eastern U. S. Because of this high cost, numerous types of brick were studied from a theoretical standpoint for suitability of physical characteristics to electric furnace roofs. Of the various bricks and combinations of bricks studied, the compound of mullite, with 71.8 pct alumina and 28.2 pct silica, combined in a stable crystal form seemed to present the best combination of properties.

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Fig. 1—Average life of mullite roofs has been 322.5 heats, with a high of 495 heats. The mullite roof shown is being removed after 350 heats.

Mullite in such purity was not available. However, there was on the market a commercial product with 62 to 66 pct alumina and 30 to 34 pct silica, with a large percentage of the total in the compound of mullite. The properties of this material, as compared with other bricks considered, are shown in Table I. The high softening point, with low linear expansion and no sudden phase change expansions, high strength at elevated temperature and high resistance to oxide penetration, all pointed to an excellent brick for electric furnace roofs. These properties combined with chemical neutrality, promised less difficulty with basic side walls and bottoms from acid spalls, and led to the decision to try mullite.

On the recommendation of the refractory company, the first trial was made with 9-in. standard bricks with a rammed center and 13½-in. electrode rings made of arch brick. The results of the first four test roofs were encouraging but not completely satisfactory. An average of 234.7 heats was obtained, with the shortest run giving 191 heats and the longest run 292 heats. Prime cause of failure was in the rammed center section where peeling of the rammed layers occurred despite all attempts to ram in such a way that layers or striations were not formed. It was noticed in all cases of failure that the standard shapes used in the outer rings were in excellent condition with 5 to 6 in. of the original 9-in. brick left at the time of failure of the center section.

Since failure occurred entirely in the rammed section and the results were otherwise encouraging, it was decided to have special shapes made up in the factory to replace the rammed center portion. Here again, failure was encountered because of peeling of the special shapes in much the same manner as the rammed centers. Results over seven roofs gave an average of 212.3 heats with a low of 166 and a high of 312 heats. One encouraging factor

Table I. Comparative Physical Properties of Various Bricks

	Mullite	Silica	Fire Clay	High Alumina
Refractoriness Softening Point	3335°F 3250°F	3090°F 2900°F	3150°F 2600°F	3250°F 2900°F
Minimum Expansion, and Contraction	Negligible expansion and contraction of extremely uniform rate 0.00000366 per °C	Extremely rapid expansion with violent rate fluctuations of different temperature ranges.	About 60 pct greater expansion and contraction with rapid changes in rate.	About 90 pct greater expansion and contraction with rapid change in rate.
Nonspalling	73 cycles under ASTM test, no defects.	10 cycles under ASTM test, 50 pct spalling loss.	10 cycles under ASTM test, 5 pct spalling loss.	10 cycles under ASTM test, 30 pct spalling loss.
Load Bearing Capacity	At 2650°F 50 lb sq in., no deformation.	At 2650°F under 25 lb sq in. load, 5 pct deformation.	At 2650°F under 25 lb sq in. load, 10 pct deformation.	At 2650°F under 25 lb sq in. load, 5 pct deformation.
Resistance to Oxides at High Temperature	Highest resistance to oxide penetration.	Fair resistance to oxide penetration.	Disintegrates rapidly due to low softening point.	Fair resistance to oxide penetration.

was the roof that gave 312 heats. It indicated the possibility of the brick if a suitable method of construction could be devised.

The refractory company was again contacted and a roof design worked out on the basis of 100 pct standard shapes, shown in Fig. 2. The ring brick series was used in the outer rings with a combination of the ring brick series and wedge bricks in the center. Twelve-in. arch bricks were used around the electrode rings. The bricks are laid up tight with a bond of mullite heat setting cement mixed into a thin slurry. The roofs are built on a cement form with a built-in heating coil. The heating coil is used to dry the cement, not to preheat the roof. It takes a bricklayer, working with a brick saw, 24 hr to build the roof, cutting all his own brick and blade using five blades per roof.

Standard brick roofs are entirely satisfactory and have more than justified the long trial period to establish the best method of using the brick. Average over 14 roofs has been 322.5 heats per roof with a

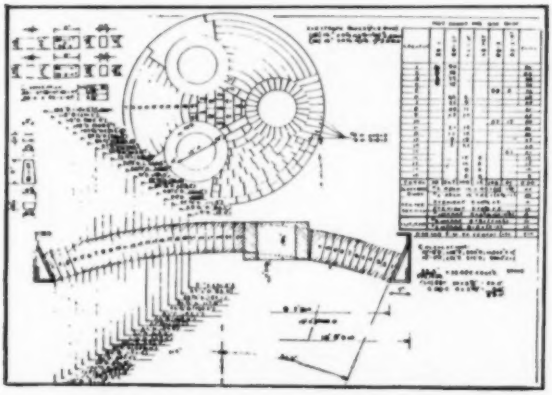

Fig. 2—Special brick shapes proved unsatisfactory, and a roof design was worked out using standard shapes. Roofs are built on a cement form with a built-in heating coil for drying the cement.

low of 229 and a high of 495 heats. The low run occurred on a roof that was damaged in an explosion. The cost of mullite roof, in the roof ring, is 1.76 times the cost of a silica roof in the roof ring.

Effect Upon Lining Life

An indirect effect of the use of mullite roofs has been increased lining life. Average basic sidewall life with silica roofs was 288.5 heats. Average sidewall life with mullite roofs has been 388.8 heats. The increase has been largely attributed to the fact that acid spalls and running of the silica roof have been eliminated with the result that basic side walls are no longer cut by the acid material. Another indirect result has been fewer roof ring repairs since the roof ring is no longer exposed by the cutting away of the top layers of basic brick by acid spalls. The lining now fails above the slag line because of reflected arc; whereas, it used to fail from the top down because of cutting by silica. It is customary to use a chrome ore parting between silica and magnesite. This was done at the Vancouver plant and operators were instructed to remove any visible spalls. Cutting caused by silica occurred despite these precautions. Chrome ore has been replaced by dolomite to form a seal between the roof ring and furnace. Operators report less bottom trouble since mullite has replaced silica, but no figures are available on this score. Door arch life has increased to the point where one replacement arch now carries the furnace through a full lining. Formerly, two replacement arches were used for an average of 100 heats less per lining. Down time for relines and roof changes decreased with a resultant increase of tons of steel produced. Full power is applied immediately after a roof change without deleterious effect upon the mullite brick. Roof change time is 1½ hr, approx.

Explosions

A point that concerned other operators and that was considered in converting over to mullite was the danger of losing a high priced roof through an explosion in the furnace. In two years of using mullite there has yet to be a roof lost through explosion. There have been a number of explosions and roofs damaged on two occasions but in both cases operations could be carried on with minor repairs and no roof change. Shortest life was 229 heats on a damaged roof. In an explosion where there is a large amount of gas formed and no projectile, the mullite roof has been strong enough to withstand the explosion. A silica roof, of necessity laid up without mortar and with expansion joints, is quite loose and will lift and separate with an explosion involving a large volume of gas. In a case where there is gas and a projectile, it was found that the projectile drives through the mullite roof and leaves a hole relative to the size of the projectile. So far these holes have not been over 16 in. diam and have been easily repaired.

Fig. 1—Finely crushed high carbon ferrochrome is mixed with silica and a binder, and then pelletized. After drying, the pellets are charged into huge furnaces where reactions take place resulting in extra low carbon ferrochrome. Operators, shown atop one of the furnaces, use a special radio system to communicate furnace readings and other data to the central control room.

Research Reaps Rewards — Huge Alloy Plant To Produce Simplex Ferrochrome, Purer Cr and Mn

TO keep pace with the overall expansion of the steel industry, and the production increase of stainless steels, Electro Metallurgical Co., a division of UC&C is engaged in a large expansion program. Part of this program was the building of a huge plant at Marietta, Ohio, costing over $100 million. This plant in addition to producing standard grades of ferrochrome, ferromanganese, and silicomanganese will produce extra low carbon ferrochrome marketed under the name Simplex, and electrolytic chromium and manganese.

Some 10 years of research, and many millions of dollars, went into the development of extra low carbon ferrochrome. That this project was successful is evident in the 7½ acres of buildings devoted to this product alone. Not only is extra low carbon (0.01 to 0.025 pct) ferrochrome beneficial to the production of low carbon (0.03 pct C max) stainless steel grades, but it is finding applications in all stainless grades. Use of this new alloy should bring about economies in the production of extra low carbon stainless steels because of faster furnace operation; and also make it possible to obtain greater overall recovery of chromium in the production of all grades of stainless steels. As is true with many things, including the A-bomb, the chemistry of the Simplex process is simple—it is the industrial production that is difficult. In Simplex, high carbon ferrochrome crushed to fine size in Allis-Chalmers ball mills is mixed with silica, and a binder, then pelletized. Under the right conditions of temperature and pressure, very high temperature and extreme vacuum, silica acts as an oxidizing agent reducing the carbon. $C + SiO_2 \rightarrow CO + Si$. The reaction takes place in huge batch type furnaces resembling autoclaves. The pellets are then removed, cooled, packed, and shipped. The plant, with four furnaces in operation and four more to be fired before the end of the year, is truly a push button operation, and walkie-talkie sets are used for fast communication. Largely automatic in character the

Fig. 2—Molten ferroalloy is tapped periodically from the electric furnaces into ladles, and then into chills where it solidifies into large flat slabs, which are crushed when cool.

entire process is controlled by special instruments in a central control room, in which operators can tell at a glance what is happening at any stage in the vast circuit of operation.

The extra low carbon grades of stainless steel were born of necessity. In stainless, it is necessary to control carbon content to prevent excess carbon from precipitating in grain boundaries and leading to corrosion. Columbium and tantalum were found to stabilize the carbon, but both elements soon became critical in supply because of their use in special high temperature alloys. Maintaining a low carbon content was found to offer the same control. Other changes have been taking place with stainless steels. The critical supply of nickel led to the development of the austenitic chromium manganese stainless steels containing 16 pct Cr, 16 pct Mn, and 1 pct Ni. The Mn was found to be a suitable substitute for Ni, although requiring almost twice as much. Now ElectroMet is setting up an electrolytic Mn plant at Marietta to produce high purity Mn in quantity. It is expected that the Mn will be available at current prices.

Electrolytic Cr will also be produced at Marietta in commercial quantities. This high purity Cr, formerly available in small quantity only, will find applications in special alloys for high temperature service and in the titanium industry.

The large amount of material produced at Marietta will be the standard grades of ferrochrome, ferromanganese, and silicomanganese. Raw materials for ferroalloy production are brought to the ¼ mile long mix house, which serves the three large electric furnace buildings. Scale cars weigh out appropriate amounts of the various materials from bins, and mixture is dumped into a skip hoist. The skip hoist travels up the inclined conveyors to the designated furnace building, where a tram car system takes over and automatically dumps each batch of mix into bins above the furnace for which it was prepared. About 1500 tons of raw materials are handled daily, resulting in 500 tons finished product.

The three furnace buildings contain 14 huge submerged arc electric smelting furnaces. These furnaces operate continuously—charge is added at regular intervals from the bins above the furnace, and the furnace is bottom tapped periodically. The alloy goes into chills, where it is cooled and solidified into large flat slabs. These slabs are crushed in the crushing bay, and screened prior to shipping.

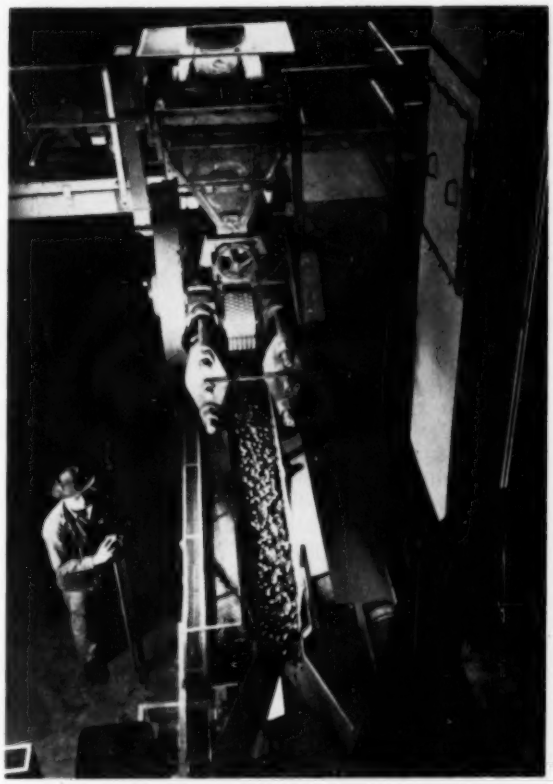

Fig. 3—Walnut sized pellets of high carbon ferrochrome-silica-binder mix are produced in this pelletizing machine. The size of the pellets enables Simplex ferrochrome to be melted rapidly after charging into a heat of stainless steel, saving furnace time.

One of the reasons for the locating of the plant at Marietta was the availability of coal from company owned holdings as well as commercial facilities. Some 40 carloads of coal a day will be used to run the plant. The Ohio River also provided a reliable source of water, as 200,000 gal of water per min are needed for cooling purposes and power. The power plant produces 200,000 kw and is one of the largest industrial plants of its type in the country.

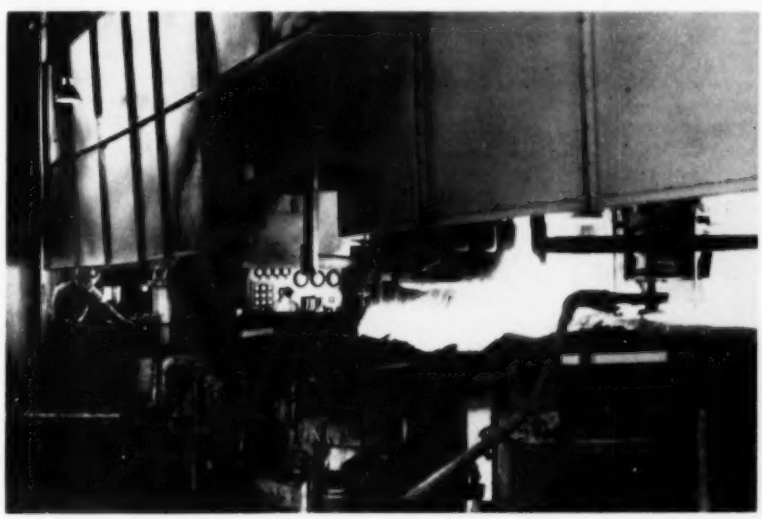

Fig. 4—On the charging floor, the operator continuously banks crushed ore, coke, steel scrap, and fluxing materials around the carbon electrodes of the submerged arc electric furnace. Furnace charges are prepared in the mix house, delivered to the furnace buildings by inclined tramways, and stored in bins above the furnaces. This is one of 14 furnaces, and will operate continuously, producing ferroalloys.

Selecting Conveyors For Handling Hot Bulk Materials

by J. Walter Snavely

PRESENT-DAY processing in many industries, calcining, sintering, briquetting, beneficiation and nodulizing, calls for handling of large volumes of hot bulk materials. For temperature ranges to 400°F, or approximately 200°C, a wide range of conveyors is available. Special constructions of rubber conveyor belts, steel conveyor belts, vibrating and shaker conveyors, apron conveyors, and drag chain conveyors, are used successfully. As temperatures go well above 400°F, however, choice of conveyors is narrowly limited. Only the problem of handling bulk materials where temperatures exceed 400°F, will be considered.

Preliminary to the study of the conveyor itself is the determination as to whether the material is to be cooled while being handled, or whether the processing requires retention of all heat and maintenance of a given temperature range.

In the majority of cases cooling is incidental to or part of the handling process; when the handling, for example, follows completion of sintering, roasting, calcining, refining, or some other process. To meet such operating conditions successfully, the conveying medium used must be fabricated to: 1—Withstand maximum initial temperatures of the material handled; 2—provide efficient heat transfer for cooling; 3—provide dependable operation and long life with minimum service requirements; and 4—provide controlled and efficient conveying.

Under usual conditions of cooling during handling, construction selected to withstand the initial maximum temperature does not necessarily involve using alloys. Excellent results can be achieved with normal carbon steels and cast irons when properly applied and proportioned.

The earliest and simplest type of conveyor for handling very hot materials is the cast steel drag chain conveyor, still widely used for handling hot cement clinker, as illustrated by Fig. 1. Because of the rugged and generous proportions of the chain link design, low carbon steels are suitable for the links. The pins, however, must be alloy steel.

The drag-chain type of conveyor has advantages and limitations. Although the efficiency of the heat transfer is relatively poor, the life of the conveyor is reasonably long, and because of its crude simplicity does not require much servicing. However, as a conveyor, it is limited in capacity, and largely limited to horizontal runs. Furthermore, the crude design, heavy weight, and the chain operating at the temperature of the material, greatly reduce permissible operating chain pulls. This type of conveyor is limited to relatively short centers.

J. WALTER SNAVELY is Conveyor Engineer, Chain Belt Co., Milwaukee. This is an abstract of an article that appeared in MINING ENGINEERING.

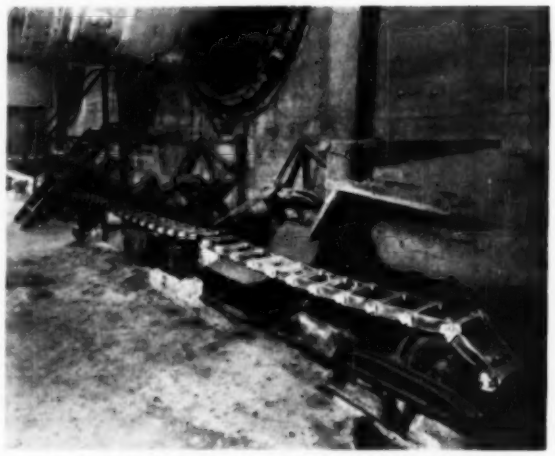

Fig. 1—Rugged construction of this cast steel drag chain conveyor permits use of low carbon steel links with alloy steel pins.

Another type of conveyor used for very hot materials is the cast pan conveyor. Because of generous proportions the cast pan, made of either cast iron or malleable iron, can withstand initial maximum temperatures. It also provides efficient heat transfer for cooling. It is on efficient conveyor construction, that can be used for inclines. Because the chain employs rolling friction instead of sliding friction, and is not in the maximum temperature zone, much longer centers are possible.

This type of conveyor is frequently used in the casting of various metal pigs, pig iron and aluminum; it is obvious, therefore, that very high initial temperatures are handled. The return run is frequently sprayed with water to accelerate heat transfer. The build-up of residual heat in the heavy cast pans is thus overcome.

The outboard roller steel pan conveyor is an improved pan conveyor that provides high rates of heat transfer and substitutes formed steel pans for the heavy cast pans. It is a very efficient conveying medium. The assembled conveyor is shown in Fig. 2, with views of both the top and the underside showing all construction details. In particular, the following general design principles were carried out in construction:

1—Capacity of the conveyor, that is, the size of the pan and the speed, together with the weights of the steels used, was designed so that the mass of the carrying pan is considerably greater than the mass of the live load of hot material. This ratio should be approximately 2 or 3 to 1 for peak load conditions. Maximum initial temperatures are easily handled, localization of intense heat is no problem.

2—Shape of the pans provide maximum surface area for efficient cooling.

3—Operating speed is low, approximately 50 fpm, permitting complete cooling on the return run and also greatly reducing the rate of wear.

4—Chain and carrying rollers are insulated from the heat zone.

5—Wear is largely confined to heavy, carrying, outboard rollers, on sleeve-type bushings, both made of heat treated white iron, extremely hard and long wearing.

6—Sole function of the chain is to pull the load, and the through-rods connecting the two strands of chain, visible in the view of the bottom side of the conveyor, further prolong chain life by providing exceptional rigidity.

7—Unique outboard roller construction permits easy servicing without disassembly of the chain or conveyor.

8—Lubrication is simple. Graphite, applied in a suitable vehicle such as kerosene, which quickly evaporates, is all that is required. Anti-friction bearings are not practical in the outboard rollers because the seals are difficult to install and maintain, and, it is difficult to retain conventional lubricants in the anti-friction bearings under prevailing heat conditions.

9—Relatively low power requirement, resulting from the use of large diameter carrying rollers, and the shape of the pans, suitable for handling up steep inclines, make this an efficient conveyor medium.

A study of the foregoing principles reveals why other types of conveyors have not proved successful for handling materials at elevated temperatures. With steel conveyor belts, either carbon or stainless steel, the ratio of the mass of hot material to the mass of the belt is high, intensifying the effect of the localization of heat. Moreover, because the belt cannot be loaded full width to the edges, temperature of the belt is not uniform across its width, setting up severe expansion problems within the belt, causing buckling at the center and subsequent cracking as it goes around the terminal pulleys.

Vibrating conveyors also have proved unsatisfactory because of poor heat transfer. The shape of the trough provides only nominal radiation area, and there is no return run for further cooling. As a result, the heat retention causes warping and distortion, which prevents the unit from conveying.

Leakproof Bucket Elevators

Where the vertical elevating of hot bulk materials is required by plant space limitations, bucket elevators can be used effectively. A specially constructed slow speed, continuous bucket elevator is in successful use handling calcined bauxite, highly abrasive, at initial temperatures of 1200°F. The bucket is entirely leak-proof in design with overlapping construction. This feature also makes possible slow operating speeds. Normal construction of continuous-type bucket elevators does not provide for overlapping buckets, and small gaps open between buckets going around the terminals. A bucket speed of approximately 125 fpm is therefore required to throw material across these gaps to prevent spillage and back-legging. With the complete overlapping construction, both on the bucket sides and on the bucket back and front, speeds of 60 fpm or slower are practical.

This bucket elevator design carries out the following general principles:

1—Capacity of the elevator, that is, the size of the bucket and speed, and the weight of the steels used provide a mass of the bucket greater than the mass of the live load being carried. This should be approximately a minimum of 3 or 4 to 1 for peak loading conditions. Maximum initial temperatures are easily handled without special provisions.

2—Design and shape of the buckets, with the extended sides, bottom and back, provide maximum surface area for efficient cooling.

3—Operating speed is low, approximately 50 fpm, permitting efficient cooling on the return run and greatly reducing wear.

4—Chain is at least partially insulated from the high heat zone.

5—Venting of the elevator casing helps to dissipate the heat.

6—Unique bucket design, entirely leakproof, keeps the chain completely clean, providing longer wear and less servicing.

7—Design is an efficient conveying medium, and can be used as an accurate volumetric feeder.

A study of the foregoing shows why the attempted use of the en masse type of conveyor has been unsuccessful for the elevating of hot materials. The chain is completely buried in the material and therefore remains at maximum temperature. The surface area is nominal, and the ratio of the mass of the hot material to the conveyor is much too high for efficient cooling. Cooling on the return run is also poor.

Additional general considerations of both the constructions described are in order. It is obvious that normal conveyor capacity ratings and ratios of live to dead loads are discarded. Providing efficient heat transfer upsets usual relationships.

Table I. Material Selection for High Temperature Use

Type of Chain	Max Temperature, °F	Heat Colors	Remarks
ASA Standard steel roller	350		
Extra clearance roller chains, replacement series roller chains	500		For best wearing qualities Nitroalloy bushings are used
Stainless steel 18-8 block or roller chains	800		Noncorrosive as well as heat resistant
	900	Faint red visible in twilight	
Monel metal block, stud or leaf chains	1000	Red visible in daylight	Nitrided pins for best wearing qualities
Cast steel chains, rollerless	1050	Blood red	Low carbon steel links alloy steel pins
Stainless steel 18-8, stud or leaf chains	1600	Bright red	
	1650	Salmon	Scaling temper 18-8 stainless
Inconel Ni-Cr alloy 80-14, stud or leaf chains	2100	Light yellow to white	A wrought material extremely scale resistant
Cast Nichrome 20-80	2100		Detachable or pintle type
Cast Chrome A 20-80	2100		Detachable or pintle type

Table II. Temperature Factors for Maximum Allowable Design Stresses, Pct

	Temperature, °F	TYPE OF CHAIN						
		A S A Standard Steel Roller	Replacement Series Roller	Rolled Monel Metal	Cast Steel Chains	Cast High Manganese Steel	Wrought Stainless 18-8	Super Alloys
MODERATE TEMPERATURES	Room 70	100	100	100	100	100	100	100
	200	100	100	95	100	89	86	100
	300	100	100	93	100	84	79	100
	350	100	100	93	100	81	76.5	100
	400		100	93	100	80	76.5	100
	500		100	92	100	76	76.5	100
	600			85	100	74	76.5	100
	700			70.5	100	74	76.5	100
	800			60	100	74	73	100
	850			54	92	74	71.5	100
	900			50.5	86	74	70	100
	950			48.5	76	74	67.5	100
	1000			46.5	64	74	63.5	100
	1050				56	74	61.5	86
HIGH TEMPERATURES	1100						56	76.5
	1200						48	59.5
	1300						37.7	47.5
	1400						25.7	37
	1500						18.6	30
	1600						15.8	25
	1700							21.5
	1800							17
	1900							12
	2000							9.5

Above data has been computed on the basis of stress causing creep rate of 1 pct elongation in 10,000 hr.

A higher ratio of mass of carrying medium to load is desirable for the bucket elevator than for the apron conveyor. The closer confinement of the elevator casing makes it harder to remove the heat and the chain is not so well protected from the maximum heat zone.

Slow operating speed increases the life of working parts far more than would normally be expected. Rather than a straight line function, with the increase in life directly proportional to the decrease in speed, the relationship is a geometric one. This is comparable to increase in life in relation to the difference of the squares of the speeds. Slowing down the operation of the units therefore pays dividends in service life.

In the design of the wearing parts, conservative unit live-bearing pressures should be used, probably only 75 pct of normal ratings. While the temperatures of the working parts are not in the critical ranges, nevertheless the temperatures are above normal, effective lubrication is largely nonexistant, and a high ratio of dead load to live load exists.

The selection of terminal equipment must likewise be tailored to the operating conditions. Such provisions as expansion joints for enclosures, gravity takeup arrangements, and bronze-bushed pillow block equipment, rather than anti-friction bearing or babbitted, are obvious necessities.

Fig. 2—Construction details of this outboard roller steel pan conveyor can be seen in the top and underside views. This type of construction permits easy servicing.

Material at Sustained Maximum Temperatures

Some processes demand sustained operation at the high temperatures. Where this condition must be met, the alloys selected must withstand the full effect of the temperatures over prolonged periods of time. Briefly, the problem is one of providing for creep, combating oxidation and loss of strength as it affects working load capacities.

Various manganese, nickel, and chromium alloys can be used for operation at elevated temperatures. Based upon extensive study and experience by the Baldwin-Duckworth div., Chain Belt Co., Tables I and II present the basis for the proper selection of alloys to be used at various temperature ranges.

Special Alloy Continuous Bucket Elevators

Outstanding examples of successful construction for sustained operation at high temperatures are the bucket elevators developed by Jeffrey Mfg. Co., and used for handling the hot catalyst in the Thermofor Catalytic Cracking Refining Process.

These bucket elevators must operate continuously 24 hr a day, 7 days a week, for a minimum of 9000 to 10,000 operating hr before any reconditioning, handling highly abrasive material at 1000°F. The elevator casings must be gas-tight to insure operation in a completely inert atmosphere. Moreover, a number of these bucket elevators are in operation handling 100 to 200 tons per hr of 50 lb per cu ft material with centers of 200 ft, in itself a real design problem. Super units will soon go into operation handling 500 tons per hr with a 300-ft lift.

Special chain was designed for proper strength, hardness, and creep rate. One detail of construction illustrates what can be done. The bushings are made of special heat-treated Ni-Hard castings, ground to size, and shrunk-fitted into the chain sidebars. At room temperature the hardness of these bushings is 600 Brinell and at operating temperatures 477 Brinell. The service life of the special chain has approached as much as 18,000 hr before reconditioning was necessary.

Phase Diagrams Play An Important Role In Powder Metallurgy

by Robert Steinitz

POWDER metallurgy has been called the metallurgy of the nonequilibrium. Phase diagrams, on the other hand, represent equilibrium; and it may be concluded that phase diagrams have no value in powder metallurgy. This, of course, is not true. All reactions that take place, for example during sintering, are always going in the direction of equilibrium. Therefore, the trend and the final state of the product will be determined by the equilibrium as represented in the phase diagram. Moreover, interest in powder metallurgy has changed in the direction of producing materials for high temperature use.

It cannot be expected that two or more materials stay out of equilibrium, if they are used at temperatures in excess of 1000°C, or if metals and ceramics are mixed together to produce the now famous cermets or ceramals. If a material is produced and tested before it has reached equilibrium, and then has to perform for several hundred hours at well above room temperature, the properties cannot remain unchanged. Equilibrium must be approached or reached, and the phase diagram will tell what equilibrium will be. The idea of taking a material, such as a carbide, boride, or oxide, and adding another material to it, usually cobalt, nickel, or copper, and expecting the second material to act as binder for the first one is correct only in a few cases. Fortunately enough, tungsten carbide and cobalt act about in this way. It is probable that just this condition made it possible to prepare cemented carbides as early as 1923. Usually the equilibrium as represented in the ternary phase diagram, e.g., W-C-Co, has to be considered, to determine if the binder really will act as such together with the hard component, or whether reactions occur changing the nature of the whole material.

The Phase Diagram

The phase diagram is based on a rule pronounced by Gibbs at the turn of the last century. One important result of this phase rule is that in binary systems there can never be a region stretching over a temperature range in which more than two phases are in equilibrium. Three phases can only exist together at one temperature, and any change in temperature will make one phase disappear. If more than two phases appear in a microstructure of a two-metal system, equilibrium cannot have been obtained; or an essential amount of an impurity is present as third component, added inadvertently through the crucible, the atmosphere, or in some other way. For three substances the maximum number of phases has to be increased by one. Only three phases can exist at the same time for a temperature range. However, fewer phases are permitted. In the binary diagram therefore, only single phase and two-phase regions can exist. Furthermore, no two single phase regions can be adjacent. They have to be separated by regions in which two phases exist.

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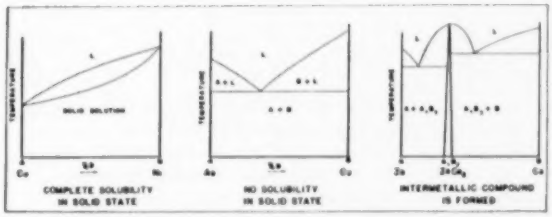

Fig. 1—There are three basic types of binary phase diagrams. In the first there is a complete series of solid solutions; in the second, complete solubility in the liquid state; and in the third, the formation of intermetallic compounds.

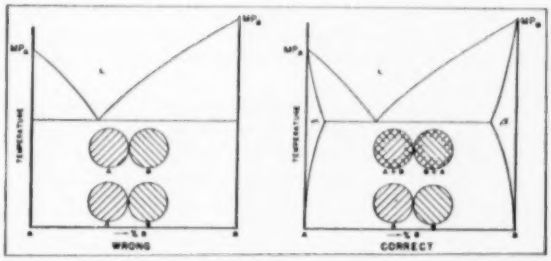

Fig. 2—To form a eutectic, some solution must occur between the two substances. The diagram at the right represents the correct formation of a eutectic.

The phase rule does not say anything about what phases occur. This has to be determined experimentally and can, for instance, be done by X-ray diffraction studies. Hausner[1] has shown how well suited powder metallurgy is for the experimental determination of phase diagrams. It is simple to prepare mixtures of two substances in any proportion. Mixtures are then heated to the selected temperature and the phases present identified by X-ray. It is important, however, that equilibrium be reached during the sintering process. Here, powder metallurgy is at a disadvantage as compared with melting. Reaction rates are considerably slower in the solid state than in the liquid, and even after hours at fairly high temperatures, equilibrium may not be obtained. Furthermore, if a large particle of substance *A* is in contact with substance *B*, the local equilibrium may differ from another point of the compact where a large particle *B* may touch a small one of *A*. If, however, equilibrium and homogenization is assured, Hausner's method is rapid and reliable for the investigation of phase diagrams.

Equilibrium means that the phases present are independent of the way in which the final state is

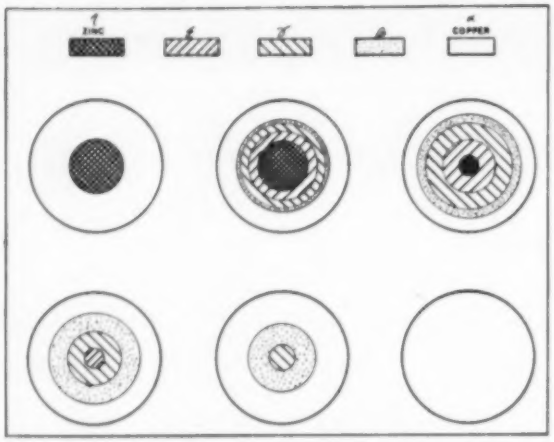

Fig. 3—During diffusion of two metals, all possible phases must occur at some time. This is a schematic representation of composition changes during homogenization of a 70-30 Cu-Zn alloy at 400°C. (Rhines.)

reached. This is important. Microscopic structures are, however, dependent on the way in which they are produced. It is impossible to obtain a eutectic structure without having been above the eutectic temperature. Bainite, troostite, spheroidal pearlite, etc., can be obtained by different treatment of the same iron-carbon composition, but all these structures consist of the same two phases, iron carbide and alpha iron.

The three basic types of phase diagrams are shown in Fig. 1. The first shows two substances *A* and *B* forming a complete series of solid solutions. This diagram might be represented by copper and nickel. The second diagram shows two substances that are not soluble in each other in the solid state, but are completely soluble in the liquid state. The third diagram shows two substances that form an intermetallic compound with each other. These intermetallic compounds can be handled in the same way as the terminal metals. The second diagram, however, is actually incorrect. If two particles *A* and *B*, e.g., of copper and silver, are considered next to each other and heated to higher temperatures, according to the diagram at the left of Fig. 2, nothing would happen until the eutectic temperature is reached. At this point a liquid phase appears. If nothing did happen during the heating up period, there is no reason why any of the substances *A* and *B* should suddenly start to melt below their melting point, as the substances have not changed. They must preserve their melting point which is much higher than the temperature at which a liquid occurs. Some diffusion and some solution of *A* in *B* and *B* in *A* must have occurred, lowering the melting point to such a degree that a liquid occurs at the eutectic temperature. The correct diagram is shown at the right of Fig. 2.

To form a eutectic—a liquid phase below the melting point of the single substances—it is necessary that some solution occur between the two substances. In the case of copper and silver, the amount is small, but still quite noticeable. In other cases the amounts might be considerably below 1 pct, but there must be *some* solution to produce a eutectic. There must also be solubility in the liquid state. If two substances are completely immiscible in the liquid state, no eutectic occurs. This is the case for copper and tungsten, but such cases are rare. Lead is another substance often not miscible in the liquid state with other metals. In the case of tungsten and copper, two particles next to each other really do not react together—there is no change in melting points. Nothing happens until copper melts at its melting point—and nothing happens afterwards either. This has the important effect that the properties of tungsten and copper are truly additive and W-Cu contacts have therefore the conductivity of copper and the hardness of tungsten. If only a slight solubility occurs in the solid state, and a eutectic is formed, then the electrical properties are considerably changed. Iron-copper compacts have conductivities much below those of either iron or copper.

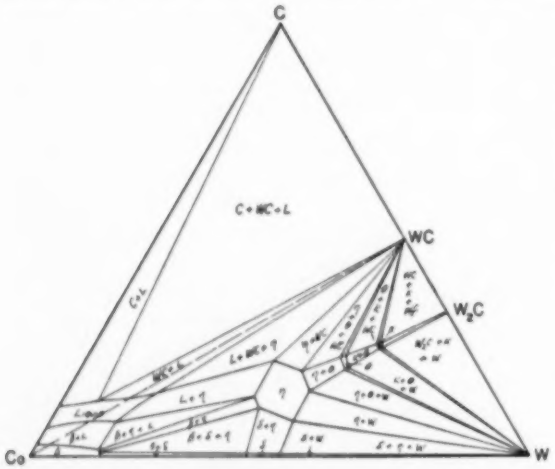

Fig. 4—This is the 1400°C isothermal section of the tungsten-cobalt-carbon equilibrium diagram. (Rautala and Norton.)

Diffusion in the Solid State, Coatings and Infiltration

Rhines has discussed the question of diffusion between two particles at higher temperature.[2] He showed that it is possible to follow diffusion by measuring properties of the compact during the sintering process. He also showed that it is possible to obtain properties of a composition not represented actually by proportion of the powder by not obtaining equilibrium during sintering. As a general rule if diffusion occurs between two particles of two metals, all the phases represented in the phase diagram must occur at one time or the other and in the order of their composition as they appear in the phase diagram, as shown in Fig. 3. They will occur at different times in different parts of the piece because of local composition differences either caused by incomplete mixing or particle size differences. If one component is very much larger in particle size than the other, the amount of intermediate phases will differ from those that occur in mixtures of equal particle size. Which phases occur can be learned from the phase diagram. Especially, it can be seen whether a liquid phase will occur at a certain sintering temperature.

The appearance of all intermetallic phases during diffusion of two pure metals also occurs if there is a protective coating of one material on another base material. If silicon is deposited on molybdenum, all molybdenum silicides which exist must appear as individual layers between silicon and the base metal. There is no possibility that in equilibrium silicon and molybdenum are next to each other. It is possible to deposit silicon on molybdenum at room tem-

perature. This layer will stay next to molybdenum at room temperature for an infinite time. This is not true because equilibrium has been reached, but because the reaction rates are so slow that equilibrium will never be reached at room temperature. Heterogeneous mixtures in powder metallurgy of two powders will behave exactly the same way. If, however, the silicon coating is to be used at considerably elevated temperatures, the preservation of non-equilibrium conditions cannot be assumed for a considerable time. What the reaction rates will be is dependent on the various substances; some might be fast, some might be very slow. It is a safe prediction that reaction rates are considerably faster at elevated temperatures than at room temperature, and it is necessary to keep in mind that as the final state only the silicide containing the lowest amount of silicon can be in contact with excess molybdenum. Even this can be the case only if the molybdenum has taken into solution all the silicon that it can absorb. The idea of having the two materials silicon and molybdenum in direct contact is not correct if equilibrium conditions will be approached.

The same conclusions hold true for the infiltration of porous powder metallurgical parts with a second lower melting metal. If the porous base metal forms compounds with the infiltrated metal, layers of these compounds have to be formed during the diffusion process at high temperatures covering all the surfaces of the porous skeleton. This might be desirable and it might be possible to stop the process at just this point when this intermetallic coating on the skeleton has been formed. In other cases, however, it might influence the properties of the material adversely. In the case of infiltration of copper into iron, no intermetallic compound is formed; however, both substances are considerably soluble in each other in a manner similar to that shown in Fig. 1 for the case of silver and copper. As the solubility of these substances is temperature dependent, precipitation hardening is possible which has been made use of to a large extent to improve the properties of the infiltrated iron skeleton. To limit the amount of iron taken into solution by the liquid copper, the infiltrated copper can be presaturated with iron. The maximum amount of iron which can be taken into solution by liquid copper can be read from the phase diagram.

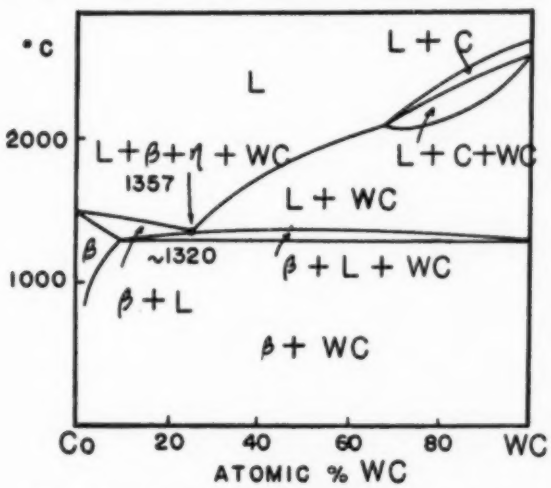

Fig. 5—Tungsten carbide and cobalt behave like two substances in a binary system. The vertical section of the WC-Co diagram shown was determined by Rautala and Norton.

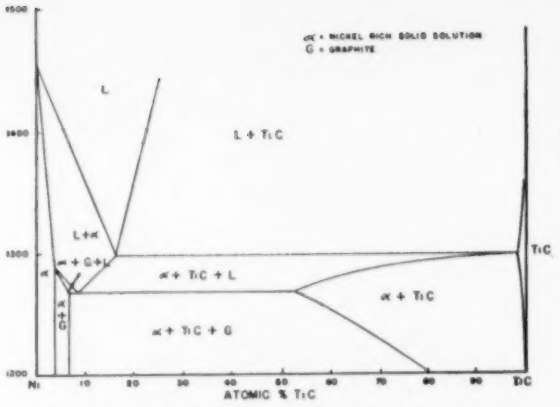

Fig. 6—The titanium carbide-nickel system has a large three-phase field, and cannot be considered as a quasi-binary system.

With these considerations in mind, the phase diagram will tell whether an infiltrant will be usable; whether intermetallic compounds are formed; whether a rapid solidification of the infiltrate will occur which might be caused by the higher melting point of intermetallic compounds.

Cemented Materials and Binders

The idea of a binder that cements another substance together is old. The classical example is the cem ted tungsten-carbide-cobalt composition. The original idea was that cobalt is added to tungsten-carbide and heated to the temperature where cobalt becomes liquid. As tungsten-carbide is much higher melting than cobalt, the liquid cobalt was supposed to run around the carbide particles, enveloping them and keeping them in place after solidification of the cobalt. This idea is incorrect. It was soon noted that it was not necessary to go above the melting point of cobalt in order to get a liquid that cements the particles together. A liquid phase occurred much below the melting point of any of the original substances. That means that reactions occur between cobalt and tungsten-carbide. Fortunately for the production of tungsten-carbide-cobalt cutting tools, this system is simple, as cobalt and tungsten-carbide behave like two substances in a binary system. The ternary diagram tungsten-carbide-cobalt shown in Fig. 4 has been investigated by Rautala and Norton,[3] and it was found that tungsten-carbide and cobalt behave like a quasi-binary system, Fig. 5. The composition does not have to be changed very much to change the picture entirely. The ternary system is necessary to understand why an undercarburized tungsten-carbide which should contain W_2C, will form the new compound eta, which is harmful in tool materials because of its peculiar properties.

The titanium-carbide-nickel diagram,[4] which is shown in Fig. 6, has a large three-phase field, so that it cannot be considered quasi-binary; only two-phase fields are permitted in a binary system. Here, the idea of a hard metal, cemented by a *binder*, is no longer applicable in this simple form. In general, it is unpredictable whether quasi-binary systems will occur, unless the ternary diagram is known.

To carry the idea of a binder a little bit further, it is obvious to try to cement one hard substance

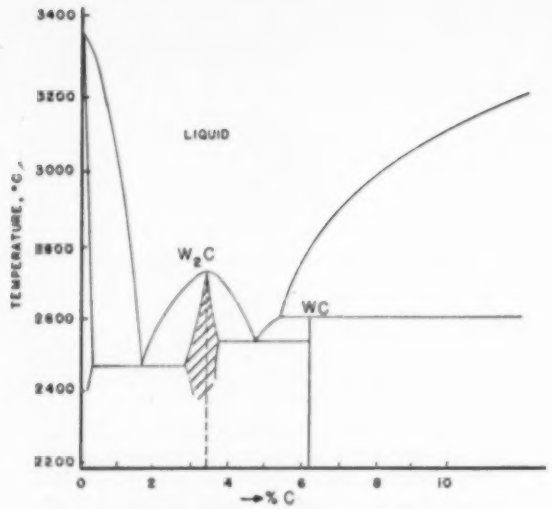

Fig. 7—Tungsten-carbon diagram shows that if tungsten-carbide was cemented with tungsten, the lowest carbide found, W_2C, would be in equilibrium with tungsten.

with its own base metal, for instance, to cement tungsten-carbide with tungsten. The phase rule requires that something like a eutectic with a lower melting point will occur between the terminal metal and its compound. This idea is correct by itself. However, tungsten-carbide is not the lowest carbide formed,[5] and can therefore never be in equilibrium with tungsten, Fig. 7. This idea of the formation of a binder by a eutectic reaction could be used only for W_2C in contact with tungsten, but W_2C has less desirable properties than WC. Therefore this idea doesn't work with tungsten-carbide. It does work, however, in a large number of other cases, but only the lowest compound can be cemented with the terminal metal. The transition metals form usually only one, and in a few cases two carbides as hard metal phases. In the case of borides this is different. Three, four, and five boride compounds are not unusual, and it is not possible to cement any but the lowest existing boride with the base metal.

If borides are to be cemented with a metal of the iron group, the situation is only in a few fortunate cases similar to tungsten-carbide-cobalt. This is, for instance, the case if CrB is cemented with nickel. A very low melting eutectic is formed. The substances in equilibrium are CrB and nickel. The eutectic occurs at about 950°C, and use of this eutectic is made in a hard facing material. The situation is different for molybdenum boride and nickel. It is possible to mix molybdenum borides with nickel, cobalt, iron or similar metals, but they do not stay boride plus metal at higher temperatures. The system MoB-nickel, as represented on the ternary diagram Mo-Ni-B in Fig. 8, shows that a new compound is formed that has the composition Mo_2NiB_2.[6] Such a compound has its own crystal structure and its own properties. Only the phase diagram can tell where it will be found, or in combination with what boride it will occur. A nickel-cemented MoB, or for that matter, Mo_2B cannot be produced. It may be possible to cement the new compound Mo_2NiB_2 with nickel, but the system is still unknown in the nickel corner.

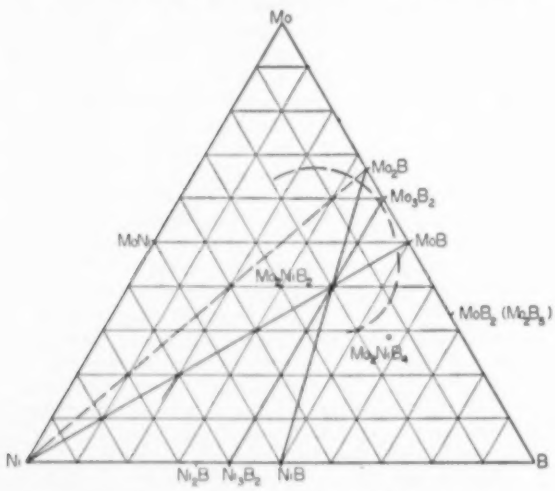

Fig. 8—Study of the ternary system molybdenum-nickel-boron shows that a nickel cemented MoB, or Mo_2B cannot be produced.

The fact that equilibrium will be the same, independent of the way in which it is reached does not mean that any one method of preparation is equivalent to another. Grain size and distribution and microscopic structure might be different but the same phases will be present. It happened that this intermetallic compound Mo_2NiB_2 proved to have interesting properties in applications as a cutting tool.[6] From the phase diagram the region of its existence is known, and it is possible to cement it together by a liquid phase produced by a eutectic between this compound and the simple boride Mo_2B.

The ideas proposed also hold true for cementing ceramic materials with metals. It has to be a fortunate chance if the mixture of an oxide with a metal remains the same after sintering as when it was mixed. Aluminum oxide-chromium mixtures have been prepared and have been declared to be valuable materials for some applications. Some of the binding action is claimed to be helped by the formation of chromium oxide and the fact that chromium oxide is isomorphous with aluminum oxide. Only the knowledge of the phase diagram will be able to tell whether the mixture chromium-aluminum oxide is in equilibrium at the temperatures at which this cermet is to be used, and it is likely that the substitution of another metal for chromium might change conditions entirely, either for better or for worse. In a similar way it is not possible to mix two hard metal phases together and request that they stay what they were at the beginning. It is not possible to mix titanium-carbide and boron carbide, and cement these substances with nickel. Reactions do occur, and titanium boride is formed. What happens with the carbon will depend on the binding metal. The unique idea of powder metallurgy, to mix any two substances together and keep them unreacted together, is not correct if it is necessary to go to temperatures, either in sintering or in service, where the reaction rates are high enough to produce equilibrium conditions.

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Further Studies of the Tuyere Zone of the Blast Furnace

by J. B. Wagstaff

The raceway in front of the tuyere of the blast furnace has been studied quantitatively and a correlation obtained for the penetration of the blast. Some evidence is presented for the height and width of the raceway which suggests that all the raceways of a furnace overlap. The size of the coke in this zone has been measured photographically during normal operation and results are given for the various areas.

IN an earlier paper,[1] it was shown that a raceway exists opposite each tuyere of a blast furnace. This raceway is formed by the jet effect of the air emerging from the tuyere and consists essentially of a turbulence in which coke particles are recirculated at high speed.

Its presence was deduced originally from observations on movies taken with a high-speed camera through the tuyeres of various furnaces and was confirmed by experiments made on a model. In the model described,[1] this raceway was shown as operating in a vertical plane only, although there was a suggestion in the motion-picture film exhibited at that time that the raceway was three dimensional, unless artificially restricted. There was also some doubt then about the factors influencing its size.

This paper describes the next steps in the investigation. Since the size of the raceway is obviously of importance in the operation of the furnace, it seemed worth while to study the subject more carefully. It is probably in this region that about half the coke in the furnace is consumed, so that the movement of the stock column may well be controlled by raceway behavior. Furthermore, there is some evidence to suggest that the coke is packed densely in the center of the furnace to form the "dead man" and more loosely above the raceway. It is therefore probable that the bulk of the gases passing up the stack flow from the top surface of this raceway. Clearly then, a knowledge of this critical zone is of interest to the blast furnace operator, and the first half of this report is devoted to a quantitative discussion of the subject.

A further topic of interest among operators is the degree of breakdown of coke in the furnace, with which is inseparably linked the importance of a strong coke. Indeed, the whole question of the optimum size and type of coke may be as dependent on the condition of the coke in the bottom of the blast furnace as at the top. Attempts have been made from time to time to obtain samples of coke from the tuyeres and other furnace openings but they all suffer from the fact that the coke is filled to a varying degree with metal and slag and is probably broken up by the very act of taking the sample. It has proved difficult to make any reliable studies of coke size by these methods.

However, it did seem possible to use the high-speed movies mentioned earlier[1] to estimate the size of the coke. These movies provide an accurate record of individual coke particles so that, in theory at least, it should be possible to measure the size of the particles one by one and to obtain, for the first time, information on the coke being blown around the raceway under actual operating conditions while the furnace is performing normally. Such a study has been made and is discussed in the second half of this paper. The results obtained enabled the blast furnace data to be correlated with the model results given in the first half.

Raceway Size

In order to make a quantitative study of the size of the raceway it was necessary to devise some apparatus of laboratory scale, which could be handled quickly and easily. This focused attention on models, which in turn means that the laws of similarity governing this particular process must be ascertained.

Method of Procedure: Since the work was to be carried out on a smaller scale than the blast furnace, the linear dimensions of the model became unimportant provided that the scale was known; it is only important to insure that the container does not affect the raceway being observed. The studies therefore were carried out in a glass-sided box, 11 in. high x 7 in. wide x 3 in. deep, using air jets ranging

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Discussion on this paper, TP 3577C, may be sent, 2 copies, to AIME by April 1, 1954. New York Meeting, February 1954; also presented at Blast Furnace, Coke Oven and Raw Materials Conference, Buffalo, April 1953.

The Transactions papers appearing in the Journal of Metals during 1953 will subsequently appear in AIME Transactions Volume 197, and may be permanently referenced to that volume.

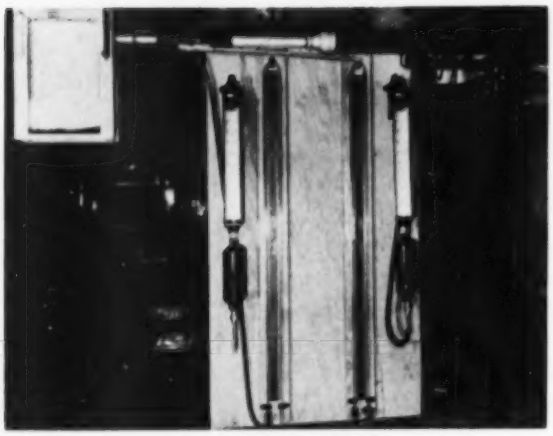


Fig. 1—Model apparatus.

from ⅛ to ½ in. in diameter. Fig. 1 shows the model box on the right-hand side filled with ⅛-in. wood particles, which served to study a single raceway. The air entered through the pipe that can be clearly seen at the bottom of the picture. Air flow was measured by readings of pressure drop across an orifice plate between the flanges shown. The orifice was calibrated against a Connersville-type positive displacement meter, proved and sealed by the Public Service Commission of New York State. The manometer used for pressure measurement is clearly shown in the photograph. The small pipe entering the box underneath the ½-in. air-supply pipe is the pressure offtake used in recording jet pressure. It was connected to the extreme end of the jet and great care was taken to insure that there was no roughness and that the pressure tap entered the jet at right angles to the jet axis.

The various types of granular materials used in the work are described in Table I. Most of the work was carried out on the wood blocks because the characteristics of the packing could then be determined with reasonable precision. The particle size was chosen so as to give the maximum practical range of size but with the limitation that it was not possible to get raceways if the particles were larger than the jet diameter, and there seemed to be a lower size limit beyond which regularly shaped particles could not be obtained. The sizes of the particles were obtained by direct measurement.

Crushed coke was used for checking the effect of particle shape. This was carefully screened to the sizes shown in Table I. The surface areas were calculated by assuming that the coke particles were all cubes and that length of the cube side was the mean of the screen sizes. This is an approximation but there does not seem to be any reliable and accepted technique of measuring with accuracy the particle surface of crushed coke. The particle sizes of the blast furnace cokes were determined by actual measurement by the method described in this paper. The particles were also assumed to be cubes.

It is realized that there may be other variables than those investigated that influence the raceway size. In particular, the packing of the particles may be important. It was found that if a raceway was blown in a freshly dumped bed by slowly increasing the air rate, there was a very sudden surge when the raceway first formed. This point was very marked but was not repeatable. If the raceway was slowly collapsed and re-formed, it started smoothly the second time and formed at a much lower air flow than before. Visual observation suggested that the difference was due to the packing of the particles. By collapsing the raceway slowly, a very loose packing was formed around the jet, which enabled the particles to start moving more easily than in the freshly dumped bed. It also seemed that the particles outside the raceway moved slightly when the raceway expanded, so that the packing density of that part of the bed at which the jet was pointed seemed to increase; at the same time, the bed vertically above the jet seemed to loosen somewhat; however, in a bed 10 in. deep, a raceway of 2½-in. diameter could be formed before there was any movement of the top of the bed. The packing density or void fraction of the bed as a whole seems to have little importance. At the same time it is not easy to measure quantitatively the void fraction of a small part of a bed.

In view of this difficulty it was decided not to study the effect of void fraction at this stage. Instead, all runs were made on freshly dumped packings. This arrangement did not simulate blast furnace conditions, but it was hoped that it would provide a simple, reproducible condition that would serve at least for the initial studies.

A rather interesting incidental question arises from the difficulty of starting the raceway. It was found that, for the same flow conditions, the pressure at the jet is much greater before the raceway is formed than afterward. If more than one jet is fed from the same air line in the model work, the tendency is for one raceway to form first, then for the air to go in large quantity to that jet, and for the other raceways not to form. This possibility, which is being studied further at the present time, raises some interesting questions about the partition of flow between tuyeres in the blast furnace.

Preliminary model studies show that the raceway becomes approximately spherical in shape if it is free to expand in all directions except backward. The particles are blown by the jet and return either sideways or over the top and down above the jet. The jet seems to have a capacity for sucking in particles where the air emerges from the metal tube. In many cases, a small secondary recirculation occurs under the jet. This seemed to be quite minor in size and was treated as part of the main disturbance.

Since the main purpose of this work was to throw light on the penetration of the blast into a furnace, the most important measurement was the maximum "diameter" of the raceway measured in a direction parallel to the jet axis. If the raceway is free to expand in all directions, it must be surrounded by

Table I. Detail of Particles Used in Model Studies

Material	Size, In.		Particle Density
	Diameter	Height	
Wood cylinders	0.130	0.124	0.674
Wood cylinders	0.2535	0.2223	0.639
Wood cylinders	0.0770	0.1092	0.700
	Screen Opening, In.		
Crushed coke	0.0331	0.0788	1.0838*
Crushed coke	0.0788	0.157	0.9184*
Crushed coke	0.157	0.263	0.9281*

* Measured by the method proposed by Ergun.[3]

the particles being studied and cannot be examined visually, so that any estimates of its size must be made by probes. This is more difficult than visual observation. The box was fitted with a wire probe ending in a ¼-in. disk, which could be pushed out above the jet until it hit the more solid bed. Sometimes it was easy to tell by feel when the probe hit the bed, but sometimes the probe would enter the bed comparatively easily, particularly when large particles and high air rates were used.

A series of measurements was therefore made to compare depth taken with the probe with those obtained visually with the jet close to the corner of the box blowing along the glass side. Thus the measurements taken with the probe could be "calibrated" against those taken visually. The jet was then moved to the center of the box side, so that the raceway could expand freely and be measured again. The results were somewhat erratic but seemed to show that the raceway was the same size whether it was restricted by the side of the box or not. Later measurements therefore were taken by visual observation. The raceway was always free to expand both upward and away from the glass at right angles to the jet.

An attempt was made to measure the size of the raceway in the horizontal direction at right angles to the jet axis by moving the jet away from the glass side until the raceway could just be detected. The method was not satisfactory because it was never certain whether the glass still interfered with the raceway formation. However, it was the only method available without cutting the raceway horizontally.

Results of Tests on Raceway Size: As this work was aimed at a study of raceway size, it is important to understand exactly what this size really is. The raceways are best pictured as roughly spherical in shape, so that they may be described in terms of three "diameters" at right angles. These three diameters are not quite the same size, so that it seems advisable to think of one as the main measurement and the other two as subsidiary. The one selected to be the main measurement was the horizontal diameter parallel to the jet axis; the other factors, such as air velocity, particle size, etc., were correlated with it.

The author was unable to find any work in the literature giving the effect of the different variables on raceway size. The work on jets, about which a great deal has been published, did not seem to apply and a theoretical approach seemed to be extremely complicated. As a result, purely empirical methods were used. However, it was found possible to group the variables into dimensionless ratios. It has been found that for many processes the grouping of variables into ratios that are independent of the units employed (as long as they are consistent) allows a wide range of conditions to be correlated. It was hoped that the same might apply in this case.

The ratios used here were

$$\frac{\rho V^2 D_j}{g \sqrt{S} P_j} \text{ and } \frac{D}{D_j}$$

where ρ is the density of air at the jet; V, the velocity of air at the jet; D, the diameter of the raceway; D_j, the diameter of the jet; g, the acceleration due to gravity; S, the surface area of an average particle; and P_j, the absolute pressure at the jet. Fig. 2 shows

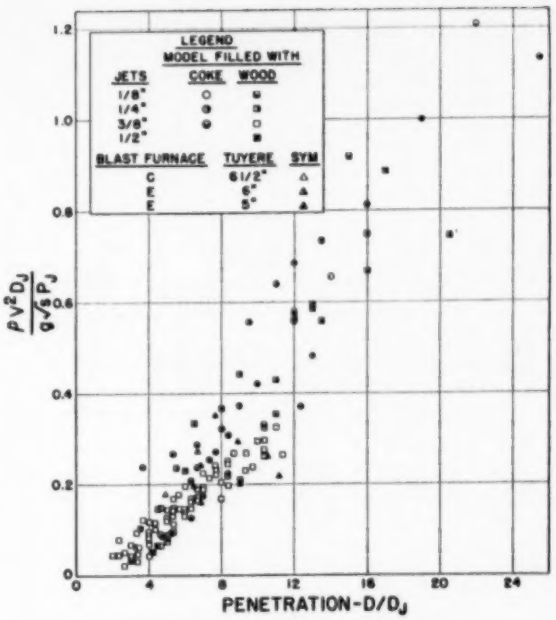


Fig. 2—Correlation of raceway diameter.

the relationship between these two ratios for the data available. This covers quite a wide range of variables because it has been found possible to include data taken on two different blast furnaces along with the data obtained from the model. The range of mean size of coke particles on the model was from 0.0560 to 0.210 in., or nearly 4 to 1, and up to 1.475 in. on the furnace, or a total range of 25 to 1. The range of wood-block size was 2.5 to 1 and the jet ratio 4 to 1 on the model, or 50 to 1 overall. All the points both from the model and the blast furnaces seem to fall fairly well in a band, as shown by Fig. 2.

In accepting the model technique as a method of studying raceway size, certain very definite disadvantages must be appreciated. The models are at uniform temperature and no chemical reaction takes place, conditions that do not occur in the blast furnace. Again, the models discussed in this paper do not involve movement of the main bulk of the bed but only of the gas. Also, there was no liquid in the models.

Despite these major differences, the correlation shown in Fig. 2 would appear to suggest that the main variables governing the raceway size are now understood. It does not seem probable that the agreement shown is a coincidence, as the correlation is reasonably complex and involves not only the flow conditions but particle and tuyere size as well as the overall pressure on the system.

It was mentioned earlier that no attempt was made to study the effect of the packing. It is almost certain that this is an important omission. Again, it can happen that the bed becomes aerated and therefore tends to expand, and the raceway size also increases. In any case, the model measurements are only approximate, as raceway diameter can be measured to the nearest particle at best and in some cases this represents a unit in the ratio of raceway-jet diameter, D/D_j. In a few cases with the ¼-in. wood blocks, raceway movement seemed gradually

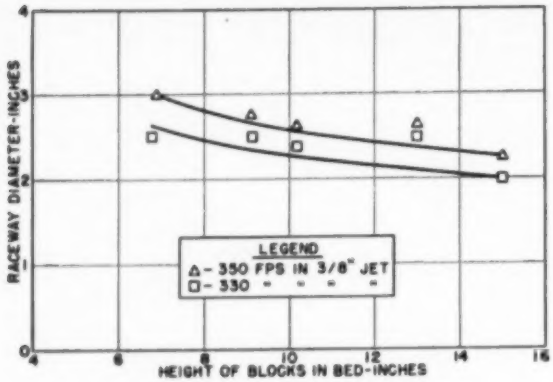


Fig. 3—Effect of bed height on raceway diameter.

to slow down into the bed and the dividing line between the rapidly moving particles and the stationary bed was indefinite. Undoubtedly this accounts for some of the spread observed in Fig. 2.

In an actual blast furnace, it can happen that the coke fails to feed smoothly into the raceway and then the void must increase, owing to combustion. This probably happened in blast furnace E and the smaller values of raceway-jet diameter, D/D_j, are therefore the more reliable.

Also the curve does not pass through the origin; but at zero flow there was a raceway of some two or three jet diameters, if extrapolation is permissible to this extent. This probably was due to the change in the packing density produced in the surrounding bed when the raceway was formed. When the air was turned off after a run, so that the raceway collapsed, the bulk volume of the bed as a whole decreased, despite the loose packing formed near the jet. That part of the bed subject to only slight movement must have compacted considerably. This observation compares with the commonly held view of a dense central zone or "dead man" in a blast furnace.

This discussion would not be complete without some reference to the many minor issues involved. For example, it was found that the height of the bed in the model affected raceway size slightly. This is shown in Fig. 3. The point was not followed in detail because somewhat different conditions exist in the blast furnace, in which there is some vertical movement of the bed. It was preferred to standardize on about 10 to 11 in. of particles, where the effect of changes of depth was slight.

While these measurements were being taken, a rather crude attempt was made to estimate the half-raceway width. The apparatus was not ideal for this purpose and the results should be accepted with caution. Fig. 4 shows the band produced by these measurements when half-raceway width is plotted against the penetration or main diameter of the raceway. It seems reasonable to draw the conclusion that the raceway is about as wide as it is long, or perhaps wider.

These measurements may be applied to the blast furnace with the precaution that there seems to be some slight change of shape as a raceway gets very big; that is, when the jet velocity is high, of the order of the velocity of sound. These velocities are not approached in the blast furnace. At high velocities, the raceway seems to become more pear shaped. Attempts have been made to sketch the probable diameter of the raceways on scale drawings of actual furnaces. It seems that adjacent raceways touch each other, or nearly so. It is difficult to imagine a pillar of stationary coke a few inches wide between two streams of coke moving rapidly in the same direction. One is therefore forced to the conclusion that the raceways do "interlock" and that there is an annular ring in a blast furnace in which the coke particles are in rapid movement. From these measurements so far obtained, this ring would seem to be about 3 ft 6 in. to 4 ft wide at tuyere level in normal operation. However, with the present skimpy knowledge, these conclusions must be regarded as tentative.

The measurement of the vertical height of the raceway was complicated by the presence of a second raceway underneath the main one. The second one is much smaller but varies in size appreciably with the particular conditions of the experiment. In general, it seems to diminish in importance as the main

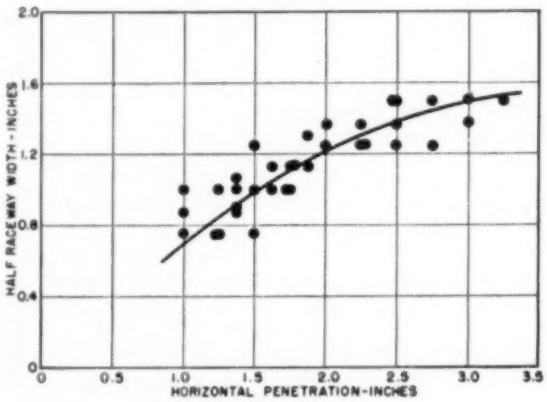


Fig. 4—Width of raceway.

raceway increases. Some evidence of this was found in all tests but so far the photographs of an actual blast furnace do not seem to show it. However, if it were there it would be difficult to detect. The second raceway could be eliminated by putting the air jet very close to the bottom of the box. It was not found in work on the narrow slice model.[1]

As mentioned earlier, it seemed best to correlate the vertical height of the raceway against the penetration rather than attempt a new correlation in terms of the air flow and other conditions. Fig. 5a shows the results obtained with wood blocks. It seemed that the larger size of particles gave higher raceways. The height did not increase as rapidly as the penetration, since the curves seem to be concave downward. During the experiment, it became obvious that the vertical height was not as precise a measurement as the others and the edge of the raceway was often difficult to determine. This was particularly true with coke particles; Fig. 5b shows the erratic nature of the results obtained. Again it seemed that the larger particles gave higher raceways.

Since the height was apparently more sensitive than the penetration to small changes of conditions, it seemed likely that it would be different in the blast furnace and in the model because the solid particles are moving down and liquids are present in the furnace. Therefore, a detailed study of this

phenomenon was left until a more complex model could be made, which would more nearly resemble the blast furnace.

Coke Size

For many years, there has been controversy about the breakdown of coke in the blast furnace, which might be settled if a method were developed for measuring the size of coke at the bottom of the furnace. Also, the correlation of raceway size includes a term involving the size of the coke being recirculated. A technique has therefore been worked out to determine the coke size in the furnace raceway without disturbing the normal operation of the furnace.

Method of Procedure: The method of ascertaining coke size consists in photographing the raceway in a blast furnace with a high-speed camera at 2000 frames per second as described earlier.[1] The film is then projected in a specially built viewer, so that the particles appear about natural size on a frosted-glass plate. A transparent plastic sheet marked with rings of known diameter is then superimposed on each particle in turn and the size is thus estimated and recorded.

The magnification was determined by measurements on the end of the tuyere, which was given about 10 pct magnification over natural size. Since the particles were about 9 or 10 ft from the camera, and the tuyere nose 8 ft from the camera, the particles could reasonably be considered about natural size.

The particles in every two-hundredth frame were counted, so that 16 frames were examined in each film. This gave about 300 to 600 particles per film. It was difficult to distinguish the individual particles when the film was stationary, but if it was moved backward and forward the particles could be located, so that the plastic sheet could be superimposed. The total movement was about 25 frames either side of the one being counted.

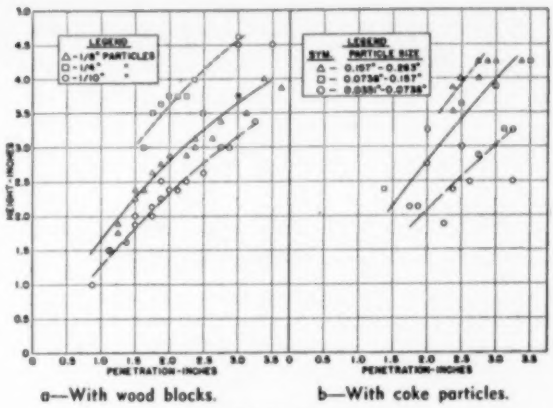
It was found necessary for two observers to work together, one measuring and one recording. Furthermore, there was evidence of fatigue if an observer measured more than two frames in succession; the observers therefore took turns at measuring. Thus two results were obtained for each film, one from each observer. The two results were compared by the chi square test and in every case the differences were found to be within that expected from normal sampling errors. In all, five observers were employed in this work and their results were in satisfactory agreement.

Since this technique is new, it is necessary to discuss its accuracy, the more so since each observer believed at first that his results were unreliable. It has so far proved impossible to devise a method of direct calibration of this technique so that indirect reasoning is the only way of assessing the inherent errors.

First, since the film is shot in 100-ft lengths and requires about 1 1/3 sec to expose, the measurements on each film represent only a very short interval of time in the life of most blast furnace phenomena. There is then some question whether this instantaneous sample is representative of furnace conditions. This question is quite different from the other question as to whether or not the results obtained accurately describe furnace conditions at the instant of measurement. At this stage, only the measurement of the particular particles viewed by the camera will be discussed.

Table II. Repeat Count of First Film Rated

	Number of Particles Observed						
Size, In.	−0.25	0.25-0.5	0.5-1.0	1.0-1.5	1.5-2.0	2.0-2.5	+2.5
1st count	57	86	139	52	17	9	1
2nd count	63	89	149	69	23	9	0
Mean	60	88	144	61	20	9	1



a—With wood blocks. b—With coke particles.

Fig. 5—Height of raceway.

The optical system is basically simple and, except for the error introduced because the position of a particular particle is not known precisely, the method would seem satisfactory. This error is reasonably small as long as great precision is not required. The biggest source of error seemed to be the ability of the observer to isolate a representative sample of the particles recorded on the film and to estimate their size. In this estimation, an attempt was made to judge whether the area of the particle shadow was greater or less than the area of the various circles marked on the plastic sheet. Then the particle could be classified as within certain size limits.

The effect of this sampling problem is difficult to check but the fact that five different observers agreed among themselves, and that size counts were repeatable within themselves, led to greater and greater confidence in the results. For example the first film rated was measured a second time some two months after the first attempt. In the meanwhile many other films had been rated and the observers had gained much experience in this difficult technique. A comparison of the results is given in Table II, showing that the repeatability is good. It will never be exact because only every two-hundredth frame on the film is counted and, despite all precautions, precise repeatability is unlikely. Further, there is some doubt that an observer measures all the particles that could possibly be measured on a particular film, thus giving additional possibility of disagreement. The main question to be answered, however, is whether the results are a reasonable estimate of the particle size recorded on the film. The answer seems to be in the affirmative, judged by all the tests applied so far.

These results are not strictly comparable with normal sieve data, which are based in essence on the technique of picking up each particle and attempting to push it through the sieve. In the film

Table III. Two Examples of the Comparison of Size Analysis on the Number Basis and the Weight Basis

Size In.	−0.25	0.25-0.5	0.5-1.0	1.0-1.5	1.5-2.0	2.0-2.5	+2.5
Furnace B							
Number, pct	1.9	10.8	42.8	28.0	11.1	4.6	0.8
Weight, pct	0.0	0.0	8.8	26.5	28.7	25.3	10.7
Furnace E							
Number, pct	40.4	23.8	25.2	7.3	2.2	0.7	0.4
Weight, pct	0.0	2.5	19.5	25.8	21.5	14.1	16.6

rating, this is obviously impossible, as the particles are observed as two dimensional shadows. However, the lack of agreement is not great enough to prevent all comparison of the two methods.

A more difficult problem to overcome arises from the way the results are recorded. The present method gives results in terms of the number of particles of a particular size present whereas the more conventional technique employs a weight basis. The two can be interconverted if it is assumed that all particles are of the same shape and density, but this is somewhat doubtful.

Another important objection to this conversion is the fact that much accuracy is lost in the process. Since one 2-in. particle weighs about the same as five hundred ¼-in. particles, a count which gives a reasonable percentage number of particles in the different categories from ¼ to 3 in. is going to show a negligibly small weight per cent of the smaller sizes. To be more specific, Table III has been drawn up to show two such comparisons. Such a conversion is obviously unsatisfactory. Thus, 40 pct by number of the smallest particles becomes a negligibly small percentage on a weight basis, whereas less than 0.5 pct of the material over 2½ in. becomes 16 pct when converted to the weight basis. The result is also sensitive to the size assumed for the oversize particles. As there seems to be no way out of this difficulty the results in this report are expressed on a number basis.

Results of Tests of Coke Sizes: As a first attempt to try out the method, a brief survey was made of the coke in the different areas of the United States. The conditions under which this series of films was taken are shown in Table IV. In most of the tests, the furnaces were on full wind. In the Southern practice, the films were shot with a 1-in. lens instead of the 4-in. lens used for the rest of this work, with the result that the magnification in the viewer was different and the appropriate correction had to be applied to the measurements taken.

The results of this survey are shown in Table V, in which it is possible to arrange the coke in order of decreasing size, with Northern the largest and Western the smallest. The difference between Southern and Western coke is not very great. This order does not differ much from that which would be expected, except possibly that the Northern coke was appreciably larger than the Eastern.

It is not easy to assess the relative differences between these various cokes, since each size analysis involves so many numbers. A test has been devised, however, to show whether different analyses vary more than would be expected because of the chance factors of sampling. This test is known as the chi square test. When it is applied to the analyses given in Table V, it shows that, if the −¼-in. size is neglected, the analyses for furnaces C, D, and E are homogeneous within each furnace but that the differences between the furnaces are real and cannot be ascribed to sampling differences. This is remarkable in connection with furnace C, where film 54 was taken 18 months before the other two. It suggests that the coke size in the tuyere zone of a furnace may be a reasonably definite and permanent quantity. While this conclusion requires confirmation, such agreement is surprising. If true, this conclusion would seem to be of major importance.

The reason the −¼-in. data have been disregarded in this statement is that in some cases small objects, which seem to be "atomized" slag, are present and it is not possible to be certain that some of these were not counted with the small coke, despite the fact that reasonable precautions were taken to exclude them. This is particularly true of Western practice. In this, the slag droplets are sometimes so frequent as to appear like a rainstorm. This was so severe that out of nine films of Western practice only three were found clear enough to rate, owing either to excessive slag or to the presence of large pieces of agglomerate which were thought to disturb the raceway. This statement might be taken to imply that conditions are worse than in fact they are. The films were taken originally to study the different furnace conditions with particular emphasis on unusual occurrences. It has not yet been possible to take a second series of films on these furnaces, designed primarily for coke-size measurements.

The data in Table V show that there is much less uniformity in the coke in the Northern practice. The difference is striking because the films on each furnace were taken on the same day, yet tests show that the size analyses cannot reasonably have come from the same homogeneous batch of coke. This difference was so marked that it was investigated in some detail. However, before discussion of that study, attention should be directed to the compara-

Table IV. Furnace Conditions During Filming

Film No.	Date	Practice	Furnace	Wind, Cfm	Blast Pressure, Lb per Sq In.	Blast Temperature, °F	Time
133	6-4-51	Northern	A	85,000	19	700	Midway between casts
135	6-4-51	Northern	A	85,000	18	710	During cast
137	6-5-51	Northern	B	72,000	13.5	730	Midway between casts
138	6-5-51	Northern	B	72,000	15	730	Midway between casts
54	6-17-49	Eastern	C				½ hr after cast
126	1-11-51	Eastern	C	91,000	21.1	1060	1 hr 18 min after cast
127	1-11-51	Eastern	C	91,000	21.1	1095	1 hr 24 min after cast
17	3-24-49	Southern	D		24		2½ hr before cast
18	3-24-49	Southern	D		12		2½ hr before cast
151	9-14-51	Western	E	60,000	28	910	½ hr before cast
154	9-14-51	Western	E	61,000	22	900	Midway between casts
158	9-18-51	Western	F	48,000	21	1100	1 min after flush, 1 min before check

Table V. Size of Coke in Different Areas

Furnace	Film No.	Plant	Size, In.						
			−0.25	0.25-0.5	0.5-1.0	1.0-1.5	1.5-2.0	2.0-2.5	+2.5
A	133	Northern	15.7	22.1	37.1	17.2	5.7	2.2	0.0
A	135	Northern	2.9	10.0	42.8	22.2	13.0	5.6	3.5
B	137	Northern	1.9	10.8	42.8	28.0	11.1	4.6	0.8
B	138	Northern	0.0	3.6	47.4	28.3	12.3	5.1	3.3
C	54	Eastern	14.5	30.4	28.9	16.3	7.9	1.8	0.2
C	126	Eastern	34.0	21.7	26.4	9.7	5.0	2.0	1.2
C	127	Eastern	32.1	23.7	23.7	13.2	3.8	2.6	0.9
D	17	Southern	25.0	23.4	30.4	10.7	7.8	2.2	0.5
D	18	Southern	25.5	24.8	29.2	9.7	7.6	2.6	0.6
E	234	Western	29.1	24.5	29.1	13.0	4.1	0.0	0.2
E	237	Western	40.4	23.8	25.2	7.3	2.2	0.7	0.4
F	239	Western	34.7	28.7	27.2	6.3	2.2	0.6	0.3

tively small amount of fine material observed. When it is remembered that the results shown are based on the number of particles, it will be realized that in this practice there is a negligible quantity of coke under ¼ in. and very little under ½ in. This is unexpected, in view of the fact that the coke is burning away in this zone. The presence of so much large coke also means that there cannot be much breakdown in the furnace except possibly of the largest material. There does not seem to be sufficient information in the literature to enable a calculation to be made of the amount of fine material to be expected at a particular time due to burning of larger lumps. The amount of fines seen, however, suggests that either the lumps break up suddenly to particles too small to be seen or that there is some

Table VI. Timing of Film Shots for Furnaces A and B

Day	Furnace A		Furnace B	
	Time	Film	Time	Film
1	10:00 a.m.	255	2:00 p.m.	258
	11:00 a.m.	256	3:00 p.m.	259
	12:00 noon	257	4:00 p.m.	260
2	2:00 p.m.	264	10.00 a.m.	261
	3:00 p.m.	265	11:00 a.m.	262
	4:00 p.m.	266	12:00 noon	263

segregation process involved, which automatically removes the small pieces. It is known that the gas velocities in this part of the furnace are high, so that it is reasonable to suppose that elutriation may take place, which would account for the phenomena observed.

On the model some runs were made with mixed 1/10-in. and ¼-in wood particles and it appeared that the 1/10-in. particles tended to flow out above the raceway, sometimes working back up the bed. On the coke runs, any fine dust formed by abrasion was blown up through the bed to the top. Often it looked as though the coke in the raceway was somewhat coarser than that in the rest of the bed; that is to say, it contained less fines. Although no measurements have so far been taken on this, all the evidence seems to support a theory of elutriation.

This preliminary survey has served to show that the size of coke in the furnace can be measured under actual operating conditions of temperature and pressure at least with sufficient accuracy to separate the cokes of the different parts of the country. These measurements are independent of the amount of slag or metal absorbed in the pores of the particles because the results are based on the number of particles. This is in contrast to any measurements based on weight. The method of measurement is extremely cumbersome and tedious but seems the the only one available. It may well prove to be of value, as so little is known of this important subject.

Detailed Studies of Coke Size in Northern Practice: In order to study the apparent irregularities of coke size in furnaces A and B operating on Northern practice, three films were taken at hourly intervals at the times shown in Table VI. During this test furnace A was supplied with −3½ +1-in. coke and furnace B with −5 + 1-in. material.

The results of the coke-size measurements are shown as histograms in Fig. 6. The sizes obtained on furnace B are larger apparently than those on furnace A, but there is considerable variation between the different ratings obtained at hourly intervals on the same furnace. Film 263 from furnace B shows an unusual size grading, as only one particle out of three hundred observed was less than ½ in. In all cases, the quantity of small material was comparatively low, as was found before. The counts of all particles less than ½ in. were grouped and no effort was made to separate the −¼ in. from −½ +¼ in. It was realized from the earlier work that the

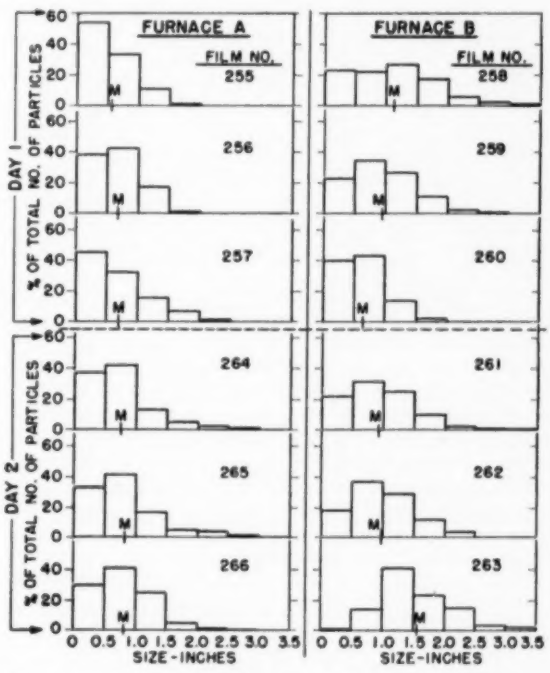


Fig. 6—Distribution of particle size on films. M indicates mean particle size.

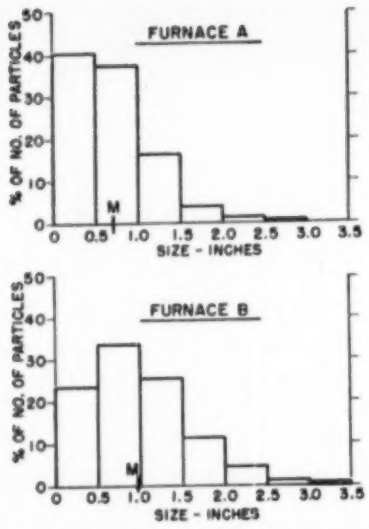


Fig. 7—Average size analyses of coke. M indicates mean particle size.

−¼-in. size was the least reliable count because of slag and other impurities.

An attempt was made to study statistically the differences in the size analyses obtained. First, the chi square test was applied to the three films taken in each of the four batches, to see whether they could be regarded as different samples from the same batch of coke. The results are shown in Table VII. The probability that the three films really described the same batch of coke is negligibly small in every case. This means that in these two furnaces the coke definitely changed from hour to hour, which did not seem to be true of the other furnaces observed. The cause of this change is unknown.

Owing to these changes between individual film counts, it is not sound to average the film analyses in each group. This greatly complicates the determination of the next problem, which is to decide whether the differences between furnaces, or between days on the same furnace, are greater than the differences between the hourly observations. Unfortunately, there is no simple statistical test to indicate whether the size gradings taken as a whole differed more between the four groups than within a particular group. It was therefore necessary to concentrate on the mean size of particles in each film.

Table VIII shows the mean size of coke in the various films. Disregarding the difference between morning and afternoon, a comparison can be made using the analysis of variance. Such a study showed that the difference between results on the different days is not larger than the difference found from hour to hour in one day. However, the difference between furnaces was real, since a difference as large as that observed would have less than one chance in twenty of being observed if there actually were no differences between furnaces. The mean sizes were 0.699 and 0.978 in. for furnaces A and B, respectively. To show the relative differences more clearly, Fig. 7 has been drawn up giving average size analyses for the two furnaces. This more detailed study confirms the fact that the coke on furnaces A and B changed considerably over short times. However, as each film is shot in about 1 1/3 sec, it is an instantaneous sampling device for most purposes. Then again, the size of the coke sample is much smaller than that usually taken. This might be thought to have an influence on the lack of consistency but in fact it probably does not. The sample used for size analysis by conventional techniques must be large because the addition of one large piece that weighs the same as several hundred small ones may seriously disturb the results of a small sample. Here one piece more or less affects the size analysis to the same degree, no matter what its size. It is therefore possible to use much smaller samples, and two or three hundred particles are sufficient to give a reliable result. The method is impractical with very small particles.

Table VII. Probability of Agreement of Size Analysis of Each Batch of Films

Furnace	Day	X^2	Degrees of Freedom	Probability of Difference Due to Chance Factors
A	1	65.5	6	0.001
A	2	20.5	8	0.01
B	1	211.8	8	0.001
B	2	185.7	8	0.001

Table VIII. Mean Coke Size

	Furnace A		Furnace B	
Day	Film	Mean Size, In.	Film	Mean Size, In.
1	255	0.555	258	1.115
	256	0.670	259	0.945
	257	0.694	260	0.653
2	264	0.739	261	0.926
	265	0.811	262	0.993
	266	0.809	263	1.551
Overall		0.699		0.978

The fact then remains that in furnaces A and B there is a considerable change in coke size from hour to hour, and possibly more frequently. The importance of this to the furnace operation cannot be accurately assessed without some information on the sensitivity of the furnace to frequent changes. Nevertheless, it would seem to be an unfortunate condition. At the present time there is no explanation of the difference between these two furnaces and the others.

Acknowledgment

The author wishes to thank the many people whose cooperation made this paper possible. In particular, R. A. Buchanan took all the high-speed movies on which the work is based. K. V. Smith and G. A. Welsch carried out much of the experimental work, R. Roeloffs gave much help and advice with the statistical work. The superintendents of the various steel plants not only cooperated by allowing measurements to be taken but also gave practical assistance and made many suggestions based on their wide experience with blast furnaces. Throughout the work B. M. Larsen gave continual encouragement and much helpful advice.

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Electrical Resistance of Titanium Metal

by James L. Wyatt

The electrical resistance of titanium as a function of purity and temperature was measured from −325° to 2800°F. Two points of inflection in the data plots were found, and an increase in resistance with increase in temperature above the transformation was observed.

IN 1948 Greiner and Ellis[1] reported on the thermal and electrical properties of titanium, including a study of the effect of temperature on the electrical resistance. The materials used in the research were obtained from the United States Bureau of Mines, where powder metallurgy techniques were being employed for consolidation purposes. These authors found a decreasing resistance with increasing temperature above 1625°F, and concluded that the latter temperature represented the point of transformation from hexagonal close-packed to body-centered cubic structure on heating. They reported a specific resistivity value of 55×10^{-6} ohm-cm, and showed that $d\rho/dt$ was positive, decreasing with increasing temperature above ambient.

In one of his early texts Hume-Rothery[2] mentioned that certain metals, including titanium, exhibited minima in plots of resistance vs temperature. He indicated that this occurred at 212°F.

Reporting many years ago on the effect of temperature on the electrical resistance of zirconium, de Boer and Fast[3] showed that phase transformation in this sister metal occurred over a range of temperatures, the extent varying with its purity. Graphs of resistance vs temperature, showing transformation effects over a range of several hundred degrees, were presented for relatively impure metal. In the latter case the resistance decreased with increasing temperature throughout the region of partial phase transformation, thereafter increasing as predicted for normal metals by modern theories of the solid state.

Since the quality of metal used in previous studies was substandard compared with iodide-type titanium, and in view of the fact that there seemed to be some rather unusual observations in the reported literature, it was decided to undertake a more extensive study of the thermal effects using high purity titanium, and to include temperature ranges beyond those which had been studied in order to ascertain whether or not the characteristics of zirconium would be duplicated.

Experimental Materials

Materials used in this investigation included commercial quality titanium (Ti-75A produced by the Titanium Metals Corp. of America) in the form of ¼ in. diam rods, and high purity iodide titanium (produced by the Foote Mineral Co.) in the form of crystal bar of irregular size. The latter was cold-swaged into rod form and machined to the desired size. Lead wires to all specimens consisted of 18 gage Ti-75A. Chemical analyses of the stock materials are given in Table I.

Experimental Procedures

Specimens 2½ to 3 in. in length were prepared with diameters nominally 0.15 to 0.20 in. Two holes,

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Discussion on this paper, TP 3550E, may be sent, 2 copies, to AIME by Sept. 1, 1953. Manuscript, Nov. 24, 1952.

This paper represents part of a thesis by J. L. Wyatt submitted in partial fulfillment of requirements for the degree of Doctor of Science to the Metallurgy Dept. of Massachusetts Institute of Technology, February 1953.

Table I. Chemical Analyses of Materials

Element	Pct	Element	Pct	Element	Pct
Iodide Titanium					
N	0.002	Al	0.012	Sn	0.003
Mn	0.01	Pb	0.007	Mg	0.001
Fe	0.004	Cu	0.001		
By difference, Ti—99.96 pct					
Commercial Titanium (Ti-75A)					
Cl	0.03	Sb	0.002	W	0.002
N	0.002	Sn	0.001	V	0.001
O	0.06	Mg	0.02	Cr	0.002
Si	0.08	Cu	0.001	Mo	0.001
Fe	0.03	Pb	0.002	Ni	0.002
Al	0.01	Mn	0.015		
By difference, Ti—99.74 pct					

sized to allow entry of the lead wires, were drilled transversely through both ends, approximately ¼ and ½ in. from each end, respectively, and the leads passed through to extend about ¼ in. The specimen surface in the vicinity of the holes was then peened to form the best electrical contact possible. Current leads were inserted in the outermost holes, the inner ones accommodating the voltage leads.

Resistance measurements were made with a Kelvin double bridge. Readings could be taken with accuracy of ±0.000001 ohm; data are therefore believed to be precise as tabulated.

Resistance values reported at temperatures below ambient were made with specimens immersed in liquid nitrogen, a dry ice-acetone mixture, or in an ice-water mixture. Temperatures below 75°F were measured with a copper-constantan thermocouple; Pt-Pt-10 pct Rh couples were employed for all other temperature determinations.

A platinum-wound alundum tube furnace with Variac control, equipped with suitable purification trains for removal of impurities from the helium atmosphere, was used for the high temperature portion of the investigation. A continuous flow of helium was maintained throughout each run. Temperatures were manually controlled, the resistance readings being taken after three consecutive temperature observations 2 min apart were found to coincide. Experience showed that this precaution probably was not necessary for the size of specimen employed, since the resistance was found to be extremely sensitive to temperature change. Readings were taken during the heating and the cooling cycles; data reported were all taken on the heating cycle, however. In every case it was observed that the readings taken during the cooling cycle were slightly higher than the corresponding readings taken on heating, with the exception that values between 1620° and 1700°F, i.e., in the range of partial transformation, lay to the left and below the values plotted.

Experimental Results

Table II is a tabulation of experimental data recorded during this investigation; in addition, the data of Greiner and Ellis, taken from a plot in their paper, are included for comparative purposes. In the table the first column lists the test temperature; the second shows the actual measured resistance for the particular specimen being used. The values listed in the third column are calculated, arbitrarily taking the value of 1.000 as the resistance of iodide titanium at 77°F. All data reduced in this manner correspond to a hypothetical specimen size which would give the value of unity to the resistance of the iodide titanium specimen; corresponding values were then adjusted for the other types of metal in accordance with the specific resistivity values of the specimen. Specific resistivity values were determined at 77°F by means of a special device incorporating two pinpoint contactors spaced a precise distance apart. The accuracy is estimated to be ± 0.02 ohm-cm. The accuracy of the specific resistivity value reported by Greiner and Ellis is not known.

Discussion of Results

The experimental data, plotted in Fig. 1, do not substantiate Hume-Rothery's statement that there exists a minimum in the curve at 212°F. On the basis of these limited amounts of data there is no indication that with increasing impurity content a curve would be obtained which would exhibit a minimum. No explanation can be offered for this reported anomaly.

It seems obvious that the conclusions drawn by Greiner and Ellis were due entirely to a lack of experimental data above 1600°F. Undoubtedly had their work been continued to higher temperatures a curve of the general characteristics exhibited by the other two curves would have resulted. On the basis

Table II. Electrical Resistance Data for Titanium vs Temperature

Iodide Titanium			Commercial Titanium			Greiner and Ellis		
Temperature, °F	Resistance, Ohms x 10^{-3}	Adjusted Resistance	Temperature, °F	Resistance, Ohms x 10^{-3}	Adjusted Resistance	Temperature, °F	Resistance, Ohms	Adjusted Resistance
−323	0.124	0.140	−323	0.331	0.250	77	0.128	1.000
−118	0.490	0.554	−116	0.841	0.623	210	0.156	1.219
+32	0.787	0.890	+32	1.240	0.918	478	0.210	1.640
79	0.887	1.003	85	1.395	1.033	761	0.258	2.016
296	1.415	1.600	388	2.228	1.648	1013	0.292	2.280
518	1.862	2.106	715	2.970	2.198	1270	0.318	2.482
923	2.500	2.828	1020	3.541	2.621	1535	0.335	2.617
1343	2.943	3.328	1305	3.937	2.914	1644	0.338	2.641
1612	3.053	3.453	1500	4.095	3.031	1734	0.325	2.539
1644	2.777	3.141	1603	4.123	3.052	1832	0.320	2.500
1675	2.774	3.137	1660	3.963	2.933	R = 55 ohm-cm x 10^{-6}		
1695	2.778	3.142	1708	3.831	2.836			
1722	2.780	3.144	1797	3.826	2.833			
1749	2.781	3.145	1900	3.852	2.851			
1786	2.783	3.148	2004	3.879	2.870			
1851	2.788	3.153	2111	3.908	2.893			
1925	2.792	3.158	2228	3.932	2.910			
2009	2.801	3.168	2420	3.972	2.940			
2090	2.816	3.185	2623	4.028	2.982			
2154	2.823	3.193	2735	4.073	3.015			
2240	2.841	3.213	R = 54.17 ohm-cm x 10^{-6}					
2374	2.850	3.223						
2563	2.860	3.235						
R = 54.98 ohm-cm x 10^{-6}								

of these data it can be stated that β titanium has the normal metal characteristic of increasing electrical resistance with increasing temperature.

In the transformation range this investigation has shown that the characteristics of titanium and zirconium are very similar. The apparent decrease in resistance with increasing temperature from 1625° to about 1700°F is due to the fact that in this region there exists not one phase but a mixture of two, probably due to small quantities of oxygen and nitrogen in the less pure varieties of titanium. The effect is almost entirely missing in the case of the iodide specimen, attesting to its purity. Both oxygen and nitrogen have the effect of raising the transformation temperature of titanium, so that the observed slope of the curve is as would be expected. Further, the relative slopes of the curves in the transformation region could be related directly to the amount of impurity present, a decreasing slope

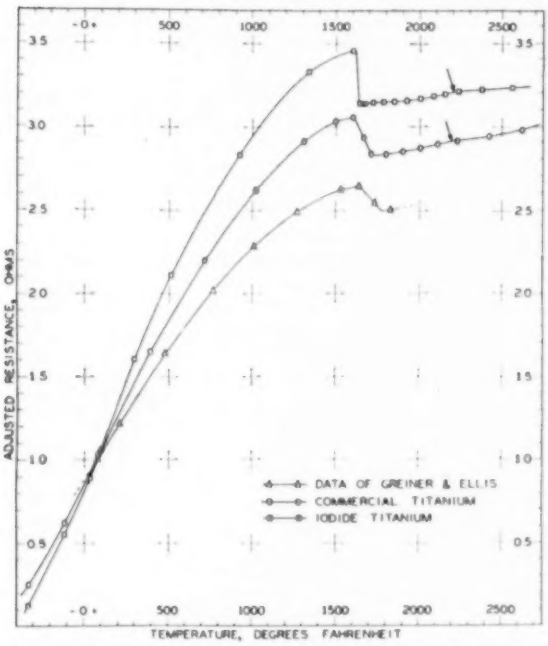

Fig. 1—Electrical resistance of titanium metal as a function of temperature.

corresponding to an increasing impurity content, qualitatively.

It is a characteristic of metals that the introduction of alloy additions increases their resistance. In this respect it would appear that titanium acts as a normal metal only at temperatures below approximately 100°F. Contrary to theory, the data indicate that the curves cross at about 100°F for various grades of titanium; the impurities are assumed to be of the same general type in each case. An additional observation shown by the data is that at the approximate point of crossing the curves undergo a point of inflection, reversing their directions of curvature. Such a phenomenon allows titanium to maintain a certain degree of respectability as a normal metal only above the point of intersection, as it is generally true that in a particular family of alloys the pure metal exhibits the maximum value of $d\rho/dt$ at any particular temperature. Unfortunately the region between −100° and +300°F was not investigated in detail, so the exact temperature at which this reversal occurs was not uniquely determined.

It may be noted that at approximately 2200°F, indicated by arrows on the curves in Fig. 1, there occurs an irregularity in the plots. No readily apparent explanation of this is evident, since it occurs in the high purity metal as well as in the commercial variety. Existing phase diagrams for titanium and the known impurities are incapable of explaining these inflections. Since the curves were reproducible by alternate heating and cooling, any effect of precipitation or grain growth was eliminated.

Conclusions

The effects of temperature on the electrical resistance of high purity and commercial purity titanium from −325° to +2800°F have been determined. A plot of the data obtained, together with data reported in the literature, has shown the following:

1—From −325° to +1625°F the resistance of titanium increases continuously, without any evidence of a minimum within this temperature region.

2—Above the temperature of completion of phase transformation the resistance of titanium changes only slightly, but does increase with increasing temperature.

3—The temperature range of transformation is a function of the impurity content and is characterized by a decrease of resistance with increasing temperature. This is associated with the simultaneous presence of α and β titanium.

4—The resistance of titanium above about 100°F is apparently decreased by the addition of certain solutes, probably oxygen and nitrogen. This observation is contrary to laws governing metals in the solid state, but is observed in such materials as germanium. In this respect titanium acts somewhat as a semiconductor.

5—The curves of resistance vs temperature for different grades of titanium were observed to cross, intersecting near 100°F, these curves presumably representing different solute contents of a single family of alloys.

6—Discovery of an irregularity in the curves at approximately 2200°F is reported.

7—The relatively high degree of purity of iodide titanium is attested to by the observed transformation characteristics.

Acknowledgments

The author would like to give special credit to Rolf Nordheim, through whose generosity the measuring equipment was made available, and to Charles Stein, who supplied the furnace for the tests. The project was under the supervision of N. J. Grant, Dept. of Metallurgy, Massachusetts Institute of Technology, and was made possible through the financial support of the Titanium Div. of the National Lead Co., Sayreville, N. J.

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Microscopical Examination of Tin Bronzes in the Alpha Range

by E. C. W. Perryman

On electropolishing, high-purity Cu-Sn and Cu-Sn-P alloys with more than 5 to 9 pct Sn were found to contain many grain boundaries with a ridge-and-furrow profile. The effect was not eliminated by solution treatment and was present at the new boundaries in recrystallized material. It could be produced by diffusing tin into the 3 pct Sn alloy and it is concluded that the effect is due to enrichment of certain boundaries in tin, or possibly in an impurity present in the tin, in solid solution.

IN the hot working of tin bronzes, especially those with moderately high tin contents, considerable difficulty is frequently encountered from intercrystalline weakness, which results in cracking at the edges in sheet on strip rolling and in the unsupported regions of the rod in rod rolling.[1] These difficulties are not at all pronounced with up to 3 pct Sn, but at 5 pct Sn impose serious limitations and at higher tin contents, especially if the phosphorus content is also high, they may become very severe. In some work carried out at the laboratories of The British Non-Ferrous Metals Research Association it was found that a notable lack of ductility in tension at elevated temperatures, associated with intercrystalline failure, was characteristic of tin bronzes even of relatively high purity. That the intercrystalline fracture is not due to a brittle or molten impurity phase at the grain boundaries was shown by the fact that the temperature at which intercrystalline fracture first occurred decreased with decrease of rate of straining. Tensile tests on some commercial tin bronzes, reported by Goetzel,[2] show a similar fall in ductility with increasing temperature.

As no visual evidence had been obtained showing that the grain boundaries of the tin bronzes were different from those in copper, a more detailed microscopical examination was carried out, and the results of this are given in the present report.

Several workers[3-6] have observed that electrolytic polishing is a powerful tool for detecting small amounts of a second phase and in particular for detecting high solute concentrations, "equilibrium segregation," at grain boundaries. Since it had been found that the suggested solutions for electropolishing bronze and the standard solutions for brass and copper did not give satisfactory results it was first necessary to find a suitable electropolishing solution.[4]

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Discussion on this paper, TP 3535E, may be sent, 2 copies, to AIME by Dec. 1, 1953. Manuscript, Oct. 14, 1952. Cleveland Meeting, October 1953.

Table I. Spectrographic Analyses of Basis Materials

Element	In Copper, Pct	Limit of Detection, Pct	In Phosphor-Copper, Pct	Limit of Detection, Pct	In Chempur Tin, Pct	Limit of Detection, Pct
As	N.D.*	0.002	N.D.	0.002	N.D.	<0.004
Sb	N.D.	0.0001	N.D.	0.0002	N.D.	<0.001
Bi	N.D.	0.0001	0.0001		<0.0001	
Sn	N.D.	0.0005	N.D.	0.0005	—	—
Pb	0.001		<0.0003		0.005	
Te	N.D.	0.004	N.D.	0.004	—	—
Zn	N.D.	0.001	N.D.	0.001	<0.001	
Cd	—	—	—	—	N.D.	0.001
Al	N.D.	—	—	—	<0.001	
Si	<0.008		<0.008		—	—
Cu	—	—	—	—	0.0013	
Ag	0.002		0.002		N.D.	—
Mn	0.001		N.D.	0.001	—	—
Fe	0.001		0.0002		0.001	
Co	N.D.	0.0001	N.D.	0.0001	—	—
Ni	0.0003		0.001		<0.001	

* N.D. = Not detected.

Experimental Procedure

Materials Used: The cast and wrought materials used for this work were the same as those used for the mechanical tests referred to above. They comprised 3, 6, and 9 pct Sn alloys without phosphorus and the same three basic alloys with 0.03 and 0.4 pct P. A 5 pct Sn alloy without phosphorus, of similar purity, was also examined.

The basis materials were Chempur tin, high purity 15 pct phosphor-copper and cathode copper, and spectrographic analyses are given in Table I.

Treatment: The cathode copper was first melted under charcoal, degassed by plunging marble chips beneath the surface, and cast through a coal gas flame into sand molds to produce a number of small ingots which after machining were of a suitable size for subsequent vacuum melting. Chill cast bars of the alloys, approximately 1¼ in. in diameter, were then made by melting and casting in vacuo, the tem-

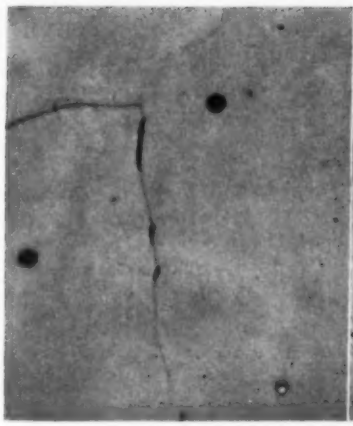

Fig. 1—As cast 6 pct tin bronze annealed at 625°C for 16 hr, water-quenched. Electropolished and electrolytically etched. X750.

Fig. 2—Wrought 3 pct tin bronze annealed at 625°C for 90 hr, water-quenched. Electropolished and electrolytically etched. X75.

Fig. 3—Wrought 6 pct tin bronze annealed at 625°C for 16 hr, water-quenched. Electropolished and electrolytically etched. X100.

perature and pressure just before casting being 1200° to 1225°C and less than 5 microns, respectively.

Analyses for nonmetallic impurities were made on typical ingots with the following results: Oxygen—0.0008 pct for bronzes without phosphorus and not more than 0.0001 pct for bronzes with phosphorus. Hydrogen—5×10^{-6} pct. Sulphur—0.002 to 0.008 pct.

The wrought alloys were cold-rolled from these cast bars, after homogenizing for 7 hr at 625°C in oxygen-free nitrogen, to bars ¾x⅜ in. The 3 pct Sn alloys without phosphorus and with 0.03 pct P were rolled directly to finished size, while the remainder were annealed at 600°C in hydrogen at approximately ⅞ in. square.

All the materials, both wrought and cast, were finally given a heat treatment of 4 hr at 625°C in oxygen-free nitrogen after which the structures were all single phase.

Specimens containing phosphorus were annealed at 625°C for 90 hr in nitrogen and rapidly quenched in water at room temperature. This temperature was chosen because it is less than the eutectic temperature (637°C) of the Cu-Sn-P system.[8] For the binary Cu-Sn alloys temperatures up to 850°C were used.

Specimen Preparation: Specimens were electrolytically polished as described by Perryman.[9] Two electrolytes were used: 1—67 pct orthophosphoric acid, 10 pct concentrated sulphuric acid, and 23 pct distilled water for alloys up to 6 pct Sn, and 2—47 pct orthophosphoric acid, 20 pct concentrated sulphuric acid, and 33 pct distilled water for alloys with tin contents of 6 and 9 pct. The polishing conditions were the same with both solutions, namely, 1.8 to 2.2 v and a current density of 0.1 amp per sq cm.

The time of polishing depended upon the state of the initial surface but starting from a surface which has been ground on emery and rough polished with metal polish, 15 min was generally found to be sufficient. Because of the tendency of the second solution to cause pitting, the polishing time was generally restricted to a few minutes. To etch the specimen after polishing the voltage was reduced to 0.8 v for approximately 30 sec; the exact time of etching depended upon the metal or alloy being etched.

Taper sections of the electrolytically polished surfaces were prepared as follows: A thin layer of nickel was plated on to the polished surface followed by a thick layer of copper. The electroplated speci-

Fig. 4—Wrought 9 pct tin bronze annealed at 625°C for 16 hr, water-quenched. Electropolished and electrolytically etched. X75.

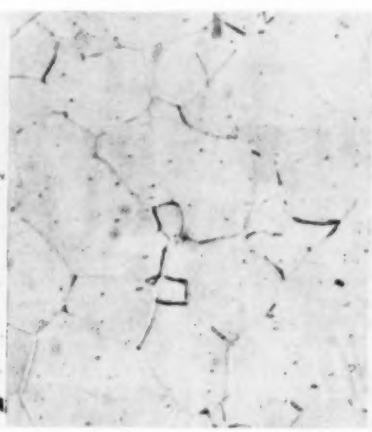

Fig. 5—Wrought 6 pct tin bronze containing 0.03 pct phosphorus annealed at 625°C for 90 hr, water-quenched. Electropolished and electrolytically etched. X100.

Fig. 6—Wrought 6 pct tin bronze containing 0.4 pct phosphorus annealed at 625°C for 90 hr, water-quenched. Electropolished and electrolytically etched. X75.

Fig. 7—3 pct tin bronze coated with tin and annealed at 625°C for 2 days. As electropolished. X1000.

Fig. 8—Wrought 9 pct tin bronze annealed at 625°C for 90 hr, water-quenched. Electropolished and electrolytically etched. X750.

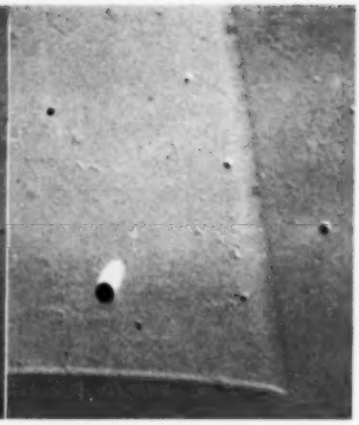

Fig. 9—Wrought 3 pct tin bronze annealed at 625°C for 90 hr, water-quenched. Electron micrograph of electropolished surface. Formvar replica Pd/Au shadowed. X10,300.

men was then placed on the inclined face of a special follower in the mounting press and the specimen mounted in bakelite in the usual way. The wedged-shape mount so obtained was then turned back in a lathe until the nickel-bronze interface was exposed. The specimen was then polished by the diamond dust technique.[7] Specimens were taper-sectioned at angles of approximately 6° or 11½° to the original polished surface. By this means, the depth of irregularities in the original polished surface, such as ridges or grooves, are magnified 10 or 5 times, respectively, in the surface of the taper section.

Results

Examination of Cast Cu-Sn Alloys Annealed at 625°C for 16 hr and Water Quenched: Examination showed that the grain boundaries in the 3 pct Sn alloy were quite normal and were revealed by differences in level between grains. With the 6 and 9 pct Sn alloys, however, parts of some of the grain boundaries appeared darker and thicker, the number of grain boundaries showing this peculiarity being greater in the 9 pct than in the 6 pct Sn alloy. Fig. 1 shows this effect in an electrolytically polished 6 pct tin bronze. To determine whether these thick portions of the grain boundary were due to a second phase associated with the alloy system, specimens which had been annealed at 850°C for 16 hr and water-quenched were examined. No significant difference was observed, the thick grain boundaries being present as before. This result strongly suggests that the grain boundary effect observed is not due to a second phase associated with the alloy system. It should be pointed out that this grain boundary effect was present after electrolytic polishing alone without etching. In general specimens were lightly etched electrolytically in order to reveal those grain boundaries which were not apparent after polishing.

Examination of Wrought Alloys: After annealing at 625°C for 90 hr followed by water quenching the grain boundary effect which was observed in the cast and annealed 6 pct tin bronze was again seen at the grain boundaries of the worked and recrystallized materials. This effect was not apparent in pure copper, nor in the 3 pct tin bronzes containing 0, 0.03, and 0.4 pct P. It was first noticed in a 5 pct tin bronze and the number of grain boundaries showing this effect increased, 1—with tin content, and 2—for a given tin content with phosphorus content. Figs. 2 to 6 show how the grain boundary effect increases with tin and phosphorus content. Similar results were obtained on specimens which had been annealed at 850°C for 32 hr and water-quenched.

Further experimental evidence for the dependence of the grain boundary effect on tin content was obtained by hot dipping specimens of the 3 pct tin bronze in molten "Chempur" tin. After annealing these tinned specimens, at 625°C for two days, the grain boundary effect was present adjacent to the tin-bronze interface, see Fig. 7.

Besides varying with solute contents this grain boundary effect also appeared to be a function of the orientation of adjacent grains for, 1—it never appeared at all the grain boundaries, 2—it was never present on twin boundaries, Figs. 3 and 8, and 3—it occurred discontinuously where twins met the grain boundary, Fig. 8.

These results thus show that the grain boundary effect is not caused by insoluble impurities, for if it were the effect would not have appeared at the new grain boundaries formed during recrystallization. It is clear, however, that it increases with increasing tin and phosphorus content and depends also on the difference of orientation between grains.

Reproducibility of Etching Effect: To see if the observed differences between the 3, 6, and 9 pct Sn alloys were due to the different solutions used for electropolishing, specimens of the 3 and 6 pct Sn alloys were electropolished in the solution normally used for the 9 pct Sn alloy. The results obtained were the same as before. The 9 pct Sn alloy specimen after examination was mechanically polished with metal polish to remove the etch. On repolishing electrolytically followed by re-examination of the same fields, the effect occurred on the identical boundaries that it did on the first polishing. A further repolishing preceded by grinding on fine emery altered the plane of polish so that the grain pattern was changed somewhat but most grains were identifiable. Again the same boundaries as before showed the effect.

Fig. 10—Wrought 6 pct tin bronze annealed at 625°C for 90 hr, water-quenched. Electron micrograph of electropolished surface. Formvar replica Pd/Au shadowed. X8000.

Fig. 11—Taper section of an electropolished 3 pct tin bronze surface. Horizontal magnification X20,000, vertical X2000.

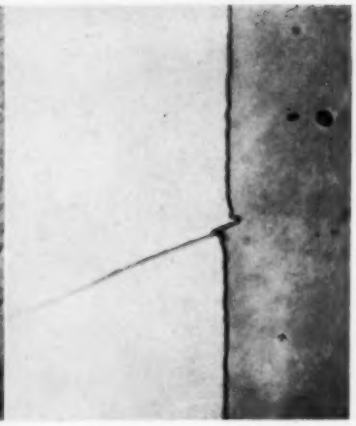

Fig. 12—Taper section of an electropolished 6 pct tin bronze surface. Horizontal magnification X20,000, vertical X2000.

Effect of Slow Cooling: Specimens of the 3 and 6 pct Sn alloys were annealed at 625°C for 90 hr, furnace-cooled at 10°C per hr to 366°C, this being 60°C above the temperature at which the 6 pct Sn alloy crosses the α-phase solubility limit.[9] After electropolishing, the 3 pct Sn alloy appeared the same as after quenching, i.e., no grain boundary effect was visible, while the 6 pct Sn alloy appeared to contain more grain boundaries showing the effect than it did after quenching from 625°C. In contrast to this a 6 pct tin bronze specimen, furnace-cooled from 850° to 450°C at 260°C per hr, appeared the same as specimens quenched from 850° or 625°C.

Electron Microscope Examination: To investigate further the nature of this grain boundary effect formvar replicas of the electropolished surfaces were prepared and examined before and after Pd-Au shadowing. Figs. 9 and 10 show typical electron micrographs from the 3 and 6 pct Sn alloys, respectively. The very black spots on these photographs are latex particles which have been used to show the direction of shadowing. Examination of the unshadowed replicas showed that there was a difference in level between the grains in the 3 pct Sn alloy, but that in the 6 pct Sn alloys, 1—there was no difference in level between grains, and 2—at some of the grain boundaries there was a raised portion. These observations together with those made on the shadowed replicas show that with the 3 pct Sn alloy there is a step at the grain boundary caused by a difference in level between the neighboring grains while with the 6 pct Sn alloy there is an elevated portion flanked by a groove, the general level of the adjacent grains being the same. Similar results were obtained with the 9 pct Sn alloy.

Taper-Section Examination: To confirm the finding of the electron microscope, 6° taper-sections of the electrolytically polished surface were prepared. Examination of the 3 pct tin bronze confirmed the electron microscope result that there was a difference in level between different grains, and moreover the angle of the step proved to be in general approximately the same as the angle between the grain boundary and the polished surface, Fig. 11. Examination of those alloys exhibiting the grain boundary effect showed that the contour across the affected grain boundaries was a hill flanked by a valley, the grains on each side of the grain boundary having the same level, Fig. 12. Another interesting observation was that in all cases one side of the hill was a continuation of the grain boundary, as shown in Fig. 12. No such contours as this were seen at twin boundaries where in general there was a slight step between the two grains in twin orientation.

Discussion of Results

It is clear that there is a close correlation between the results of this microscopic examination and the mechanical behavior of the alloys, since the number of grain boundaries showing the grain boundary effect increases with tin content and for a given tin content with increasing phosphorus content in just the same way as does the incidence of grain boundary weakness at high temperatures.

There are obvious difficulties in explaining the metallographic observations in terms of a second phase of the Cu-Sn system or of an insoluble impurity because: 1—the grain boundary effect was observed in alloys quenched from temperatures far away from any two-phase region in the equilibrium diagram, 2—it occurred in alloys made from high purity materials, 3—the effect was still present at the grain boundaries after recrystallization, and 4—no coalescence took place after long annealing times or after very slow cooling.

On the other hand it is possible to explain the occurrence of this grain boundary effect by the theory of "equilibrium segregation"[10] according to which solute atoms, differing considerably in size from the solvent atoms, tend to segregate to grain boundaries. The segregating solute is considered to be tin (see preceding section on Wrought Alloys and Fig. 7), although it could possibly be an impurity contained in the tin used. This possibility is remote as the main impurity, lead, was only present in the tin to 0.005 pct. The theory explains why the grain boundary effect is a function of the relative orientation of neighboring grains, for clearly the tin concentration at the grain boundaries will be dependent upon this orientation difference and in particular at a twin boundary where there is a perfect fit between the two crystal lattices there should be little or no

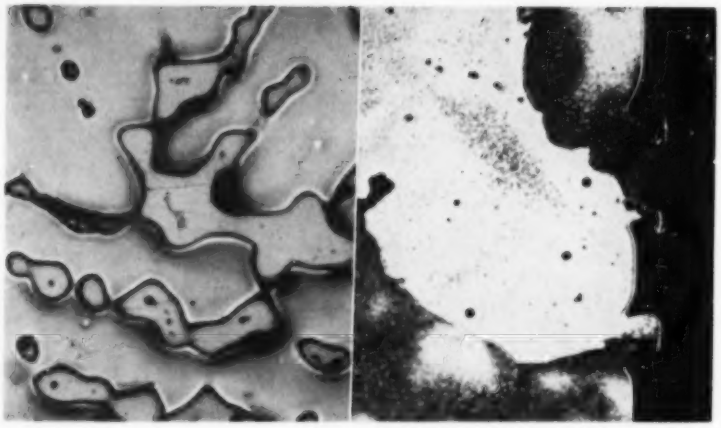

Fig. 13 (left)—As cast 6 pct tin bronze electropolished. X250.

Fig. 14 (right)—Taper section of an electropolished as cast 6 pct tin bronze surface. Etched with alcoholic ferric chloride. Horizontal magnification X1250, vertical X250.

tin concentration. Likewise the grain boundary concentration will increase with tin content, maximum concentration occurring when the grains are saturated. Similarly, in the absence of precipitation, the tin concentration at the grain boundaries should be greater at low temperatures than at high, thus explaining the increase in number of grain boundaries showing the grain boundary effect after very slow cooling. While it is clear from the results that the number of grain boundaries showing this grain boundary effect increases with increasing phosphorus content the reason for this is not certain. It may possibly be because of the effect of phosphorus in reducing the solid solubility of tin in copper, e.g., at 600°C the solid solubility of tin in copper is 15.5 pct and is reduced to 10 pct by the presence of 0.4 pct P.[5]

Similar grain boundary contours to that shown in Fig. 12 have been found by Samuels[4] in Cu-Bi alloys and by McLean[5] in Cu-Sb alloys, both authors attributing this etching effect to solute concentrations at grain boundaries. McLean held that if this contour were produced by a second phase at the grain boundaries it would be expected to be symmetrical about the grain boundary rather than asymmetrical. To show that this grain boundary contour could possibly be formed as a result of a tin concentration at the grain boundaries, a 6 pct tin bronze was examined as cast after electropolishing. It was found that while those areas poor in tin had polished, the tin-rich areas had not, the original polishing scratches still remaining, Fig. 13. An 11½° taper section of this electrolytically polished surface was prepared, and it was clearly seen that the tin-rich areas (dark areas in Fig. 14) were standing proud of the general surface level (Fig. 14), and furthermore that the rate of polishing was more rapid at parts adjacent to the tin-rich zones. Thus under the conditions of polishing used the tin-rich areas are cathodic and the tin-poor areas anodic. When two electrodes are immersed in direct contact the current density at a point on the anode will decrease with increasing distance from the cathode (see Evans[11]). This effect is occurring during electropolishing since there is a shallow trough each side of the tin-rich zones. It is clear therefore that under the conditions of polishing used there are large electrochemical differences between the tin-poor and tin-rich zones in the cast tin bronze, and it is therefore to be expected that a tin concentration at a grain boundary will make the grain boundary cathodic to the grain during electropolishing leading to a valley as in Fig. 12. From these considerations it might be supposed that owing to the cathodic nature of the grain boundary there would be a valley produced each side, resulting in a symmetrical notch rather than the asymmetrical contour shown in Fig. 12. The precise explanation of these contours is extremely difficult because: 1—the cathodic grain boundary zone is only supposed to be a few atoms thick, and 2—the electropolishing process is superimposed on the complicated system of local electrodes already present. In view of this no detailed explanation is offered. There are two features which are rather surprising: 1—the disappearance of the differences in level between adjacent grains when the effect shown in Fig. 12 occurs, and 2—the breadth of the peak on the left of the boundary in Fig. 12, bearing in mind that the postulated zone rich in tin, presumed cathodic to both adjacent grains, is only likely to be of the thickness of a few atoms. It is interesting to note that in Al-Fe alloys, which under certain conditions suffer grain boundary attack when immersed in 10 pct hydrochloric acid or 0.30N sodium hydroxide, there is the same tendency for the difference in level between grains to disappear as the grain boundary attack becomes more marked. Also in this case where there is no superimposed anodic attack, a symmetrical notch is formed at the grain boundaries.[12]

Acknowledgment

The author is indebted to the Director and Council of The British Non-Ferrous Metals Research Association for permission to publish this paper.

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Grain Boundary Attack on Aluminum in Hydrochloric Acid And Sodium Hydroxide

by E. C. W. Perryman

The wide grooves formed at the grain boundaries when high purity aluminum is attacked by hydrochloric acid or sodium hydroxide have been attributed by earlier workers to the high energy of the grain boundary material. The effect has been investigated for high-purity Al-Fe alloys with up to 0.055 pct Fe as a function of iron content and heat treatment. It is shown that the explanation given above is untenable, but that the results can be explained on the assumption that iron segregates to the grain boundary in solid solution.

IN 1934, Rohrmann[1] showed that aluminum of 99.95 pct purity suffered intercrystalline corrosion when immersed in 10 to 20 pct hydrochloric acid, and that the susceptibility to intercrystalline corrosion depended upon the heat treatment given. The greatest susceptibility was found for specimens quenched from a high temperature (600°C) and the lowest susceptibility for specimens cooled slowly from that temperature. Lacombe and Yannaquis[2] have shown that super-pure aluminum (99.9986 pct) annealed at 600°C suffers intercrystalline attack in 10 pct hydrochloric acid and that this attack is intensified by anodic dissolution in the same solution at a current density of 10 milliamperes per sq dm. No difference in extent of intercrystalline attack was found between the 99.993 and 99.986 pct Al, which led the authors to suggest that impurities played only a secondary role in the mechanism of intercrystalline corrosion. It was found, however, that the attack at the grain boundaries depended upon the relative orientation of the grains, large differences in orientation favoring rapid attack. Boundaries where the two neighboring grains were similarly orientated showed high resistance to attack as did boundaries between grains which were in twin relationship. These observations led Lacombe and Yannaquis to suggest that the intercrystalline attack was due to lattice discontinuities present at grain boundaries. Assuming that the grain boundary is a layer three to five atoms thick and has a crystal structure which is a compromise between the two neighboring grains it is clear that the discontinuities will increase with increasing difference in orientation between the neighboring grains and hence the increasing tendency to intercrystalline attack. Roald and Streicher[3] investigated the effect of heat treatment of aluminum alloys ranging in purity from 99.2 to 99.998 pct on the corrosion resistance in 20 pct hydrochloric acid and 0.30N sodium hydroxide. They found that in hydrochloric acid the intercrystalline attack appeared to be determined by the type and quantity of impurities present and by the relative orientation of the grains. No difference in the susceptibility to intercrystalline attack was observed between specimens quenched and those furnace cooled, from 575°C. In 0.30N sodium hydroxide some materials exhibited intercrystalline attack, this taking the form of V-notches.

Rohrmann[1] offered no explanation for the greater susceptibility to corrosion of material quenched from 600°C. It seems possible that this difference is connected in some way with a different distribution of impurity elements in the quenched and slowly cooled specimens. The fact that Roald and Streicher[3] observed no difference between quenched and slowly cooled specimens may possibly be due to differences in either rate of cooling or silicon content or possibly both. Both these would be expected to have an effect on the distribution of impurity elements.

Although the rate of cooling used by Rohrmann was slightly more rapid than that used by Roald and Streicher the position cannot be clarified because Rohrmann does not give the silicon content and Roald and Streicher give the silicon contents of only a few of their alloys.

That Lacombe and Yannaquis[2] found no difference in corrosion behavior attributable to impurities between the two materials they used may be because both were of high purity compared with the aluminum used by Rohrmann.[1] Although they found no difference in the corrosion behavior of their two materials it is possible that the results obtained by Lacombe and Yannaquis may, nevertheless, have been influenced by impurity distribution, since, on the transition lattice theory of grain boundary structure, it would be expected that sparingly soluble impurities would tend to segregate to boundaries where the orientation difference is such that there is a greater density of atomic sites of suitable size to contain them.

It was considered worth while, therefore, to examine the corrosion properties of a series of materials of differing impurity content with the objects of confirming the experimental observations made

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Discussion on this paper, TP 3514E, may be sent, 2 copies, to AIME by Dec. 1, 1953. Manuscript, Oct. 14, 1952. Cleveland Meeting, October 1953.

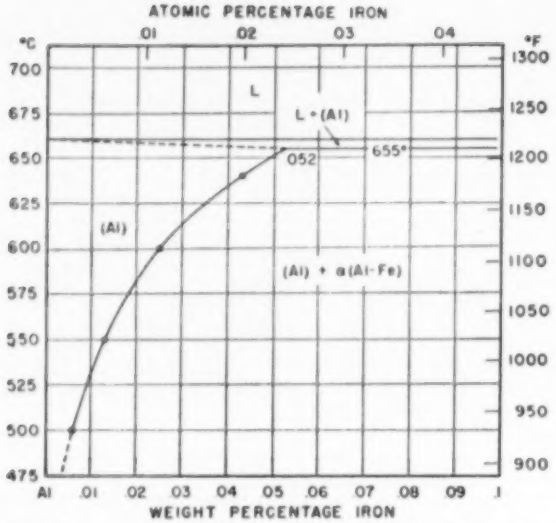

Fig. 1—Solvus curve at the aluminum end of the Al-Fe system. From J. K. Edgar.[4]

by Rohrmann, by Lacombe and Yannaquis, and by Roald and Streicher and determining whether it was possible to provide an explanation for them in terms of impurity distribution, special attention being paid to the role of iron.

In the papers referred to above it is clear that the attack at the grain boundaries occurs over a comparatively wide zone and this type of corrosion will be called "grain boundary attack" to distinguish it from the deep, penetrative intercrystalline attack found in alloys such as Al-Mg and Al-Cu.

In the Al-Fe equilibrium diagram at the aluminum-rich end, the eutectic composition has been reported to be at about 1.7 pct Fe, the phases in equilibrium with the liquid being α solid solution and $FeAl_3$. The solid solubility of iron is very small, various investigators giving the solubility as 0.028 to 0.052 pct at the eutectic temperature 655°C. The most recent investigation of this system has been carried out by Edgar[4] who showed by electrical resistivity measurements that the solid solubility of iron at 655°C was 0.052 pct. This author stated that an alloy containing 0.025 pct Fe, annealed at 640°C for 24 hr and water-quenched showed no second phase. The equilibrium diagram given by Edgar is shown in Fig. 1.

Experimental Procedure

The materials used were in the form of 0.064 in. sheet, and the analyses are given in Table I. All materials received a final cold reduction of 50 pct. It will be noted that the silicon contents are substantially the same (except for OOB1) and with the same exception the copper contents are low and all of the same order. The first five materials can therefore be regarded as essentially a series of dilute Al-Fe alloys of constant impurity.

Table I. Analyses of Materials

BNF Mark	Fe, Pct	Si, Pct	Cu, Pct
OCD	0.001	0.005	0.001
OCE	0.009	0.005	0.001
OCF	0.021	0.006	0.0005
OCG	0.037	0.007	0.001
OOB3	0.016	0.007	0.003*
OOB1	0.055	0.016	0.008*

* Determined by spectrographic analysis.

Specimen Preparation: Specimens 1 in. sq were emeried, polished with metal polish, and then annealed at 640°C for 72 hr. Some specimens were rapidly quenched in water at room temperature, some slowly cooled to room temperature in 17 hr, and others slowly cooled to room temperature in 70 hr, the approximate rate of cooling for the latter being 8½°C per hr. The specimens were then again polished on metal polish, electrolytically polished in a solution containing 1 part of 70 pct perchloric acid and 5 parts alcohol, washed in water and acetone, and dried. For the corrosion tests they were immersed vertically in 10 pct hydrochloric acid or 0.3N sodium hydroxide at room temperature.

Results

Examination of Electrolytically Polished Surfaces: The results of the microscopical examination of the various alloys are given in Table II. Typical structures of alloy OCG (0.037 pct Fe) after annealing at 640°C for 72 hr followed by quenching and by furnace-cooling are shown in Figs. 2 and 3, respectively.

Fig. 2—Al-0.037 pct Fe (OCG). Three days at 640°C, water-quenched. Electrolytically polished. X75.

Fig. 3—Al-0.037 pct Fe (OCG). Three days at 640°C, furnace-cooled to room temperature in 70 hr. Electrolytically polished. X75.

Fig. 4—Al-0.001 pct Fe (OCD). Quenched from 640°C, corroded in 0.30N NaOH for 3 days. X8.

Fig. 5—Al-0.009 pct Fe (OCE). Quenched from 640°C, corroded in 0.30N NaOH for 3 days. X8.

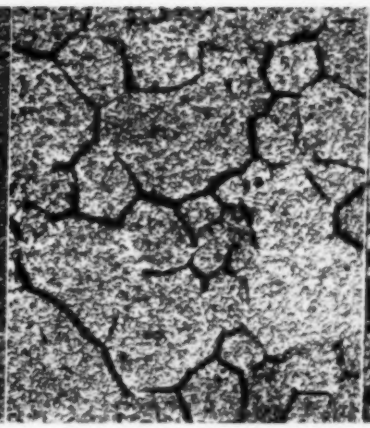

Fig. 6—Al-0.021 pct Fe (OCF). Quenched from 640°C, corroded in 0.30N NaOH for 3 days. X8.

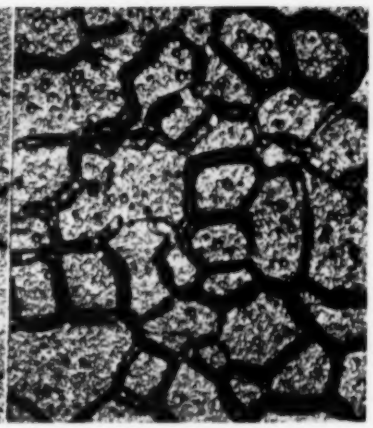

Fig. 7—Al-0.037 pct Fe (OCG). Quenched from 640°C, corroded in 0.30N NaOH for 3 days. X8.

These findings are apparently not in agreement with those of Edgar[4] who found that the solubility of iron in aluminum at 655°C was 0.052 pct. This discrepancy may possibly be accounted for by the fact that the materials used by Edgar[4] contained only 0.002 to 0.003 pct Si, while alloy OCG contains 0.007 pct Si, as well as 0.037 pct Fe. It may be that the second phase observed here is the ternary Al-Fe-Si phase and not $FeAl_3$.

Examination of Electrolytically Polished Surfaces after Immersion in 0.30N Sodium Hydroxide: Six specimens of each of the alloys marked OCD, OCE, OCF, and OCG in two conditions of heat treatment, 1—3 days at 640°C, water-quenched, and 2—3 days at 640°C, furnace-cooled to room temperature in 70 hr, were completely immersed in 0.30N sodium hydroxide for 3 days. During this time they suffered severe general corrosion, most of the specimens losing approximately 50 pct of their weight. Examination of the specimens showed that in those which had been water-quenched from 640°C the number of grain boundaries which suffered grain boundary attack and the depth of such attack increased with increasing iron content, Figs. 4 to 7. In the specimens of low iron content, which showed little or no grain boundary attack, the grain structure could be seen because of differences in level between the grains. This is presumably a result of the rate of corrosion being a function of crystallographic orientation. No such differences in level were observed with specimens of higher iron content, which suffered severe grain boundary attack.

Specimens furnace-cooled from 640°C, except those of alloy OCD (0.001 pct Fe), showed very different corrosion behavior from those that had been water-quenched. Alloy OCD showed the differential grain attack mentioned above with no grain boundary attack, after furnace-cooling as after quenching. Alloy OCE (0.009 pct Fe) which showed only little grain boundary attack in the quenched condition, showed very heavy grain boundary attack after furnace-cooling, Figs. 5 and 8. On the other hand alloys OCF and OCG (0.021 and 0.037 pct Fe) showed a very marked decrease in grain boundary attack compared with those specimens which had been water-quenched, Figs. 7 and 9. These observations are summarized in Table III.

The grain boundary attack never occurred at all grain boundaries and, moreover, was not always continuous along a single grain boundary. To investigate this further, corroded specimens were electroplated with copper and sections were polished. Examination of these sections showed that the grain boundary attack on the quenched specimens increased in depth with increasing iron content and took the form of V-notches, these V-notches being symmetrical only when the grain boundary was perpendicular to the sectioned surface, Figs. 10 and 11. Sometimes, when two grain boundaries were close together, the grain boundary groove took the form

Table II. Microscopical Examination of Electrolytically Polished Surfaces

BNF Mark	Fe, Pct	Si, Pct	Heat Treatment		
			3 Days at 640°C, Water-quenched	3 Days at 640°C, Furnace-cooled to Room Temperature in 17 Hr	3 Days at 640°C, Furnace-cooled to Room Temperature in 70 Hr
OCD	0.001	0.005	α solid solution.	—	α solid solution.
OCE	0.009	0.005	α solid solution.	—	α solid solution.
OCF	0.021	0.006	α solid solution with very few particles of a second phase. No particles of second phase at grain boundaries.	—	α solid solution with particles of a second phase. Particles of second phase larger than in the quenched material and some precipitated at grain boundaries.
OCG	0.037	0.007	Same as for OCF except rather more second-phase particles.	—	Same as for OCF except rather more second-phase particles.
OOB3	0.016	0.007	α solid solution.	α solid solution with particles of a second phase.	—
OOB1	0.055	0.016	α solid solution with particles of a second phase. More particles of second phase than OCG.	α solid solution with second phase. Second-phase particles more numerous and larger than in quenched material.	α solid solution with second phase. Second-phase particles more numerous and larger than in material furnace-cooled in 17 hr.

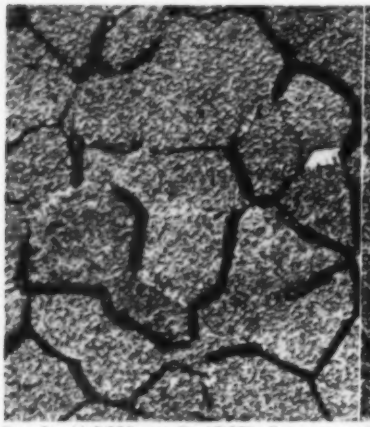

Fig. 8—Al-0.009 pct Fe (OCE). Furnace-cooled (70 hr) from 640°C, corroded in 0.30N NaOH for 3 days. X8.

Fig. 9—Al-0.037 pct Fe (OCG). Furnace-cooled (70 hr) from 640°C, corroded in 0.30N NaOH for 3 days. X8.

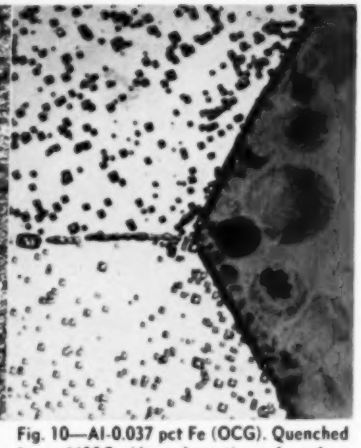

Fig. 10—Al-0.037 pct Fe (OCG). Quenched from 640°C. Normal section of surface corroded in 0.30N NaOH for 3 days. Etched in 1/3 HCl, 1/3 HNO_3, 1/3 H_2O, 2 pct HF. X200.

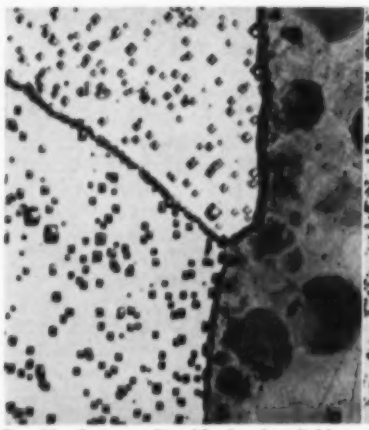

Fig. 11—Same as Fig. 10. Another field. X200.

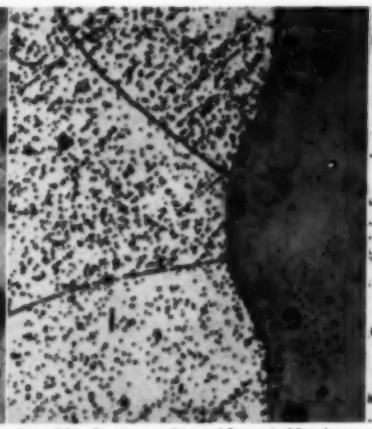

Fig. 12—Same as Figs. 10 and 11. Another field. X100.

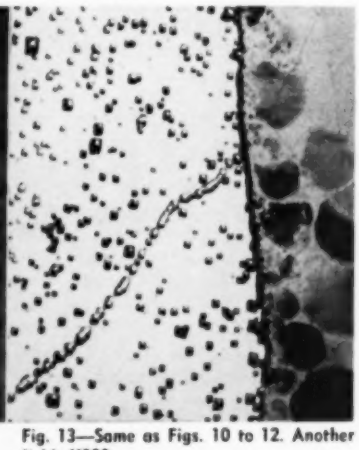

Fig. 13—Same as Figs. 10 to 12. Another field. X200.

shown in Fig. 12. It was also clear that the grain boundary attack was a function of the orientation of the neighboring grains. Figs. 10 to 13 show sections which have been polished and etched with a solution containing 1/3 hydrochloric acid, 1/3 nitric acid, 1/3 water, and 2 pct hydrofluoric acid, in order to develop etch pits. It is clear from these photographs that the grain boundary attack is dependent upon the orientation of the grains adjacent to the grain boundary.

Examination of Electrolytically Polished Surfaces after Immersion in 10 Pct Hydrochloric Acid: Alloys OCD, OCE, OCF, and OCG: Three specimens of each alloy marked OCD, OCE, OCF, and OCG in two conditions of heat treatment, 1—3 days at 640°C, water-quenched, and 2—3 days at 640°C, furnace-cooled to room temperature in 70 hr, were completely immersed in 10 pct hydrochloric acid for times varying from 5 to 40 days. As with the specimens immersed in sodium hydroxide the number of grain boundaries in the water-quenched specimens which showed grain boundary attack and the depth of such attack increased with increasing iron content, Figs. 14 and 15. The depth of grain boundary attack also increased with time of corrosion. The only differences between immersion in sodium hydroxide and hydrochloric acid appeared to be: 1—that alloy OCD (0.001 pct Fe) while showing no grain boundary attack in sodium hydroxide showed a small amount in 10 pct hydrochloric acid, and 2—the width and depth of the grain boundary grooves were much greater after immersion in sodium hydroxide than in 10 pct hydrochloric acid.

Furnace cooling, instead of quenching, had little effect on alloys OCD (0.001 pct Fe) and OCE (0.009 pct Fe) but markedly reduced the grain boundary

Table III. Examination of Specimens Corroded 3 Days in 0.30N Sodium Hydroxide

BNF Mark	Fe, Pct	3 Days at 640°C, Water-quenched	3 Days at 640°C, Furnace-cooled to Room Temperature in 70 Hr
OCD	0.001	No grain boundary attack; differential attack from grain to grain.	No grain boundary attack; differential attack from grain to grain.
OCE	0.009	Some grain boundary attack; 5 pct of grain boundaries attacked. Differential attack from grain to grain.	Heavy grain boundary attack; 90 pct of grain boundaries attacked. No differential attack from grain to grain.
OCF	0.021	Grain boundary attack; 60 pct of grain boundaries attacked. No differential attack from grain to grain.	Some grain boundary attack; 30 pct of grain boundaries attacked. A little differential attack from grain to grain.
OCG	0.037	Heavy grain boundary attack; 90 pct of grain boundaries attacked. No differential attack from grain to grain.	A little grain boundary attack; 5 pct of grain boundaries attacked. Differential attack from grain to grain.

Table IV. Depth of Grain Boundary Attack after 34 Days in 10 Pct Hydrochloric Acid

Alloy	Depth of Attack at Various Grain Boundaries, Micron
Al-0.016 pct Fe (OOB3)	3.6, 2.0, 3.0, 3.4, 2.4.
Al-0.055 pct Fe (OOB1)	4.0, 7.4, 6.4, 4.2, 4.0, 7.4, 8.8, 6.2.

attack in alloys OCF and OCG (0.021 and 0.037 pct Fe), particularly in the latter, Figs. 15 and 16.

As with the specimens in sodium hydroxide the grain boundary attack seemed to be a function of the orientation difference between adjacent grains and also the intensity of attack varied along the length of some of the grain boundaries.

Alloys OOB3 and OOB1: After immersing quenched specimens of alloys containing 0.016 pct (OOB3) and 0.055 pct (OOB1) Fe in 10 pct hydrochloric acid for a few hours small isolated pits could be seen at some of the grain boundaries. This pitting appeared first in the less pure material, and is shown in Fig. 17. After longer times of immersion the grain boundary attack became more marked and at the same time general corrosion revealed the grain structure. As before the grain boundary attack took place mainly at those boundaries where orientation difference between the neighboring grains was large, and the attack was not always continuous along one grain boundary. More grain boundaries suffered attack and the depth of attack was greater on the less pure OOB1 than on OOB3. Table IV gives a number of measurements of the depth of grain boundary attack made by focusing on the top and bottom of the grain boundary grooves. The depths varied considerably at different points on the same specimen.

To investigate the effect of furnace-cooling, specimens of alloy OOB1 were annealed at 640°C for 3 days, some were water-quenched, some furnace-cooled to room temperature in 17 hr, and some furnace-cooled to room temperature in 70 hr. These specimens were then totally immersed in 10 pct hydrochloric acid for 40 days. The quenched specimens suffered heavy grain boundary attack, approximately 95 pct of the grain boundaries being attacked. Specimens which had been furnace-cooled in 17 hr were similar to the quenched specimens except that slightly fewer grain boundaries had been attacked but specimens furnace-cooled in 70 hr were very different in that only about 10 pct of the grain boundaries were attacked. These results for the quenched and more slowly cooled material agree with those obtained with alloy OCF (0.021 pct Fe), described above.

Fig. 14—Al-0.001 pct Fe (OCD). Quenched from 640°C, corroded in 10 pct HCl for 40 days. X16.

Fig. 15—Al-0.037 pct Fe (OCG). Quenched from 640°C, corroded in 10 pct HCl for 40 days. X16.

Fig. 16—Al-0.037 pct Fe (OCG). Furnace-cooled (70 hr) from 640°C, corroded in 10 pct HCl for 40 days. X16.

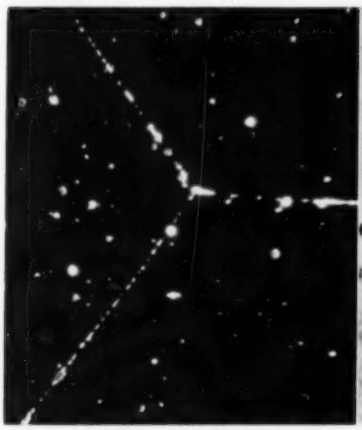

Fig. 17—Al-0.016 pct Fe (OOB3). Quenched from 640°C, corroded in 10 pct HCl for 21 hr. Dark field illumination. X250.

Fig. 18—Al-0.055 pct Fe (OOB1). Quenched from 640°C. Normal section of surface corroded in 10 pct HCl for 34 days. Etched in 1/3 HCl, 1/3 HNO_3, 1/3 H_2O, 2 pct HF. X1000.

Fig. 19—Same as Fig. 18. Another field. X1000.

Examination of sections of the corroded surfaces confirmed that the depth of grain boundary attack depended upon the relative orientation of the neighboring grains, see Figs. 18 and 19. The shape of the grain boundary grooves was similar to those found in specimens corroded in sodium hydroxide. Comparison of Figs. 10, 11, and 19 shows clearly that not only is the attacked grain boundary region much wider in 0.30N sodium hydroxide than in 10 pct hydrochloric acid, but the ratio of the width to the depth is greater.

Discussion of Results

Properties of Grain Boundaries—Attribution of Observed Effects to "Equilibrium Segregation" of Iron in Solid Solution: Before considering the results in detail it will be worth while considering the properties of grain boundaries in a homogeneous metal or alloy. Owing to the distorted nature of the grain boundary it is clear that in a pure metal some atoms will be in compression while others will be in "holes" which are too large for them. For a solid solution the resulting local elastic stresses can be reduced if large atoms are placed in large holes and small atoms in small holes. On the other hand, a solute atom in the lattice of a grain, if much larger or smaller than the solvent, will cause elastic stresses around it. Thus the energy of the system as a whole should be reduced if some of the large solute atoms are transferred from the lattice of the grain to the large holes at the grain boundary, and some of the small atoms to small holes at the grain boundary. In the equilibrium state there should thus be a segregation of large or small solute atoms or both at the grain boundary, the degree of segregation depending upon the relative orientation of the neighboring grains. This will be referred to as "equilibrium segregation." Bragg and Nye[5] have demonstrated this effect by means of a bubble model. The effect can also be deduced from Cottrell's theory[6] of solute "atmospheres" around dislocations. Experimental evidence for "equilibrium segregation" has been given by McLean and Northcott[7-9] for temper-brittle steels and antimony in copper and brass, Samuels[10] for bismuth in copper, and Perryman[11, 12] for aluminum in β brass and tin in α Cu-Sn alloys. The difficulties in obtaining direct evidence for these concentrations are considerable and the experimental evidence given by these authors is of an indirect nature. The most direct evidence for this type of segregation has been given by Chalmers and others,[13] who showed, using radioactive tracers, that polonium, which is the decay product of Bi^{210}, segregated at the grain boundaries in Bi-Pb alloys. The difference in atomic size between solute and solvent necessary for "equilibrium segregation" to occur is not known but it is reasonable to suppose that a difference in apparent atomic diameter or Goldschmidt atomic diameter in excess of about 10 pct would cause a considerable effect. Bearing this in mind, it is probable that iron in aluminum will undergo "equilibrium segregation" (Al 2.85A, Fe 2.52A). Bearing in mind that the solid solubility of silicon in aluminum is considerable and that the apparent atomic diameter of silicon in aluminum is 2.73A, "equilibrium segregation" of silicon is somewhat improbable.

In an alloy the grain boundary concentration of solute at a given temperature will increase with increasing concentration of solute within the grains. Maximum concentration will occur when the grains are saturated and any further addition of solute will result in precipitation of the next phase.

The effect of temperature on grain boundary segregation can be assessed approximately from its effect on the solid solubility. In general, when a metal contains another metal of very different atomic diameter there is marked increase of solubility with temperature. This is especially so with Al-Fe alloys, see Fig. 1. This indicates that a given concentration in the crystals is accommodated much more readily at high temperatures, so that as the temperature increases there will be a lower concentration in the grain boundaries for a given concentration in the grains. Thus for an alloy of a given composition, which is initially a solid solution, the grain boundary concentration is likely to increase with a decrease in temperature until the solid solubility line is reached. Any further fall in temperature is likely to decrease the grain boundary concentration because of the general decrease in solubility.[7] This decrease in grain boundary concentration will be accompanied by precipitation of a second phase. Thus by furnace-cooling an alloy in which grain boundary segregation is present, the grain boundary concentration will increase as the temperature falls until precipitation of the second phase starts, and with a further fall in temperature the grain boundary concentration will decrease. The extent to which these changes will take place will obviously depend upon the rate of cooling.

From the results of the corrosion tests in 0.30N sodium hydroxide and 10 pct hydrochloric acid it is clear that the rate of grain boundary attack is a function of: 1—the iron content, 2—the relative orientation of the neighboring grains and, 3—the rate of cooling from a high temperature.

The effect of iron content on the susceptibility to grain boundary attack can be simply explained by the theory mentioned above, for if the grain boundaries possess a higher iron concentration than the grains there is likely to be an electrochemical difference between them. That Lacombe and Yannaquis[8] found no difference in susceptibility to grain boundary attack between aluminum of 99.993 and 99.9986 pct purity might be because the iron contents were not sufficiently different. It is interesting to note that Montariol et al.[14] have recently found by autoradiography a segregation of impurities at the grain boundaries in aluminum of 99.998 pct purity.

The variation in degree of grain boundary attack with the orientation of the neighboring grains is also in keeping with the theory of "equilibrium segregation" because, as explained above, the amount of iron segregated at the grain boundaries will depend upon the relative orientation of the neighboring grains.

As already explained the effect of furnace cooling will be first to increase the iron concentration at the grain boundaries as the temperature falls and then when a second phase begins to appear to decrease the grain boundary concentration. Accordingly the following observations are all in keeping with the theory of "equilibrium segregation": 1— alloy OCE (0.009 pct Fe) was more susceptible to grain boundary attack after furnace-cooling than after quenching, and 2—alloys OCF and OCG (0.021 and 0.037 pct Fe) were more susceptible when quenched than after furnace-cooling when a second phase was precipitated and, 3—alloy OOB1 (0.055 pct Fe) decreased in susceptibility to grain boundary attack in the order water-quenched, furnace-cooled in 17 hr, furnace-cooled in 70 hr.

It is difficult to see how the results given here can be accounted for by the theory given by Lacombe and Yannaquis, for if the grain boundary attack is

due to lattice discontinuities at the grain boundaries: 1—there should be no difference between quenched and furnace-cooled material of very high purity, and 2—if the grain boundaries were anodic to the grains a more concentrated form of attack would be expected.

Mechanism of Corrosion: It has already been pointed out that the grain boundary corrosion observed here is very different from that observed in Al-Mg, Al-Cu, Al-Zn, and Al-Zn-Mg alloys after heat treatments conducive to such attack. It is now fairly well established that when these alloys are susceptible to intercrystalline corrosion there is a phase at the grain boundaries which is anodic to the grains. Under these conditions the corrosion at the grain boundaries penetrates deeply into the metal but is confined to a very narrow grain boundary zone, and this is in keeping with the presence of small anodes at the grain boundaries with large cathodes around them. In contrast to this, the attack observed here occurs in a relatively wide grain boundary zone; in 0.30N sodium hydroxide the width of the grain boundary groove is greater than the depth, while in 10 pct hydrochloric acid the width is approximately equal to the depth. Mears and Brown[15] found that the grain boundaries of 99.986 pct Al water-quenched from 620°C were anodic to the grains in 20 pct hydrochloric acid, while in slowly cooled specimens they were cathodic. It is difficult, however, to conceive of an anodic grain boundary leading to the shape of grain boundary grooves shown in Figs. 10 to 12 and 19. On the other hand such grooves are consistent with the grain boundary being cathodic to the grains, and indeed, if the iron concentration at the grain boundaries is greater than that within the grains, the grain boundaries would be expected to be cathodic in the corrodants used. As shown by Evans[16] the current density will, under these conditions, decrease as one goes further away from the cathodic grain boundary, the distribution of corrosion being as shown diagrammatically in Fig. 20. From this it would be supposed that the contour of the grain boundary groove would not show any sharp discontinuity with the specimen surface as it does in Figs. 10 to 12 and 19.

A number of explanations could be devised to account for the shape of the grain boundary grooves, and the variation in the ratio of width to depth of these grooves. As none of these explanations can be put forward with assurance it would not be profitable to discuss the matter in detail. It should be mentioned, however, that Fig. 20 applies to a surface free from films, and Evans[17] has pointed out that the presence of an oxide film may modify the distribution of corrosion, and possibly lead to the observed sharp discontinuity between the contour of the groove and the specimen surface. There is evidence that oxide films persist when the specimens are immersed in the acid solution (there being no visible hydrogen evolution except on scratching, a change of potential in the anodic direction on scratching, etc.). On the other hand there is little evidence that oxide films persist in alkaline solution (when there is continuous general evolution of hydrogen, little change of potential on scratching, etc.). Apart from these detailed considerations, however, the main point is clear—the grain boundary grooves produced are consistent with the presence of a cathodic zone at the boundary, i.e., with the presence of an increased concentration of iron.

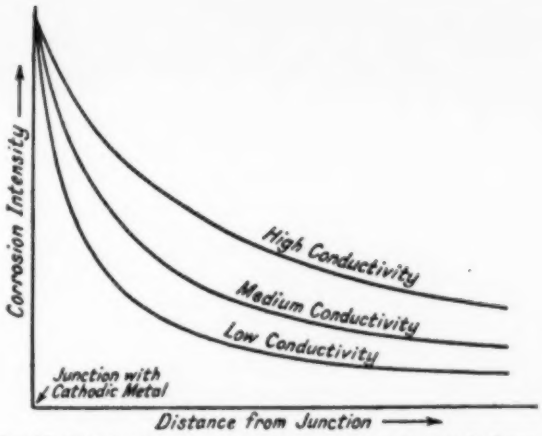

Fig. 20—Distribution of corrosion near a junction. From U. R. Evans, ref. 18, p. 530. Reprinted with permission of Edward Arnold & Co.

It was mentioned earlier (see Table III) that when only a few or none of the grain boundaries were attacked there were differences in rate of corrosion from grain to grain but that these differences were no longer present when a large proportion of the grain boundaries suffered attack. In all cases, however, it should be remembered that general corrosion was going on all the time. Presumably these differences in the rate of corrosion of different grains are due to potential differences set up because of differences in crystallographic orientation. When, however, a large proportion of the grain boundaries contain concentrations of iron, it must be presumed that the difference in electrochemical properties between these grain boundaries and grains are large enough to swamp the effect of smaller differences between the grains.

Acknowledgments

The author is indebted to the Director and Council of The British Non-Ferrous Metals Research Association for permission to publish this paper.

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Martensite Nucleation in Substitutional Iron Alloys

by John C. Fisher

Nucleation theory is applied to martensite nucleation in substitutional iron alloys. Composition fluctuations are neglected, and a steady rate of nucleation is predicted for any composition and temperature. The maximum rate of nucleation (as a function of temperature) is shown to be measurable only for an extremely narrow composition range, being too great or too small outside this range. A number of experimental observations, including completely isothermal transformation in some alloys and composition-dependence of M_s temperature in others, are compared with the calculated behavior.

IN a previous report,[1] nucleation theory was applied to the formation of isothermal martensite in an Fe-Ni-Mn alloy. The experimental observations of Cech and Hollomon[2] were accounted for quantitatively. It now is of interest to examine the predictions of nucleation theory with respect to martensite transformation in substitutional iron alloys of other compositions. Since Fe-Ni alloys have received considerable attention, both as to their transformation kinetics and their free energies, they will be treated in detail, although the results apply with equal validity to other substitutional alloys.

In substitutional alloys, where composition fluctuations can be neglected in volumes of critical nuclear size, the rate of nucleation of martensite in subcooled austenite is[1]

$$\bar{n} = N\nu \exp(-W^*/kT) \qquad [1]$$

where the free energy of formation of the critical size nucleus is

$$W^* = 8192\,\pi\,(\theta^2\sigma^3)/27\,\Delta f_v^2 \qquad [2]$$

In the expressions for n and W^*, the symbols have the following meanings: N is the number of atoms per unit volume; ν, the atomic vibration frequency; θ, a function of the elastic constants of austenite and the shear angle of martensite; σ, the austenite/martensite interfacial free energy; and Δf_v is the free energy change per unit volume accompanying the transformation.

Jones and Pumphrey[3] have determined the austenite to martensite free energy change as a function of nickel content and temperature for Fe-Ni alloys. Their expression is

$$\Delta F = 2500\,C + 1.25\,(1-C)\,\Delta F_{v_o} \text{ cal per mol} \qquad [3]$$

where C is the atom fraction of nickel, and ΔF_{v_o} is the free energy change per unit volume for pure iron as tabulated by Zener[4] (or, equivalently, Fisher[5]).

From Eqs. 1 to 3, it is possible to calculate isothermal nucleation rates as a function of temperature and composition for Fe-Ni alloys. The only unknown parameter is $(\theta^2\sigma^3)$. The best value of this parameter for Fe-Ni alloys, determined from M_s temperatures in a manner to be described shortly, is

$$(\theta^2\sigma^3) = 9.92(10)^{21} \text{ cgs units} \qquad [4]$$

Taking this value of $(\theta^2\sigma^3)$, C-curves for isothermal nucleation were calculated for Fe-Ni alloys of several compositions, and are plotted in Fig. 1.

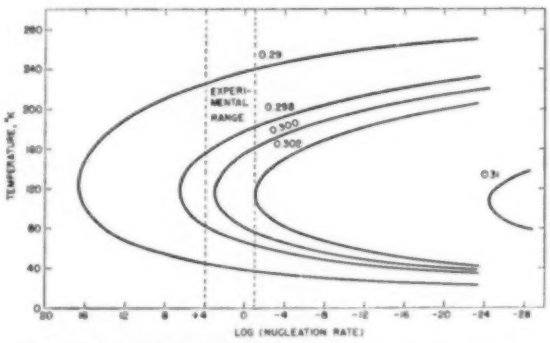

Fig. 1—Calculated isothermal nucleation rate as a function of temperature for Fe-Ni alloys in the composition range $0.29 \leqq$ atom fraction Ni $\leqq 0.31$. Rates less than 10^{-1} and greater than 10^{4} nuclei per cc per sec are difficult to measure experimentally.

It is evident from Fig. 1 that completely isothermal nucleation of martensite in Fe-Ni alloys can be observed experimentally for only a very narrow range of compositions. Cech and Hollomon find that a nucleation rate of about 10^4 nuclei per cc per sec is as fast as they can follow dilatometrically, and a rate of about 10^{-1} nucleus per cc per sec is about the slowest that can be measured conveniently. If the nucleation rate is to lie between the limits $10^{-1} \leqq \dot{n} \leqq 10^4$, the nickel content of an Fe-Ni alloy must fall in the atomic fraction range $0.299 \leqq C \leqq 0.302$. Nickel contents lower than 0.299 correspond to nucleation rates too great to follow, and those higher than 0.302 to nucleation rates too slow to measure in a reasonable time.* In view of the narrow composition range in which successful isothermal measurements can be made, it is not surprising that only one substitutional alloy has been described[2] that comes close to being in the proper range.

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Discussion on this paper, TP 3548E, may be sent, 2 copies, to AIME by Dec. 1, 1953. Manuscript, Nov. 13, 1952. Cleveland Meeting, October 1953.

* The actual nickel composition corresponding to the middle of the range may differ by 0.01 or 0.02 from the calculated 0.300, owing to uncertainties in the calculation. However, these uncertainties have little influence on the calculated width of the range.

The alloy used by Cech and Hollomon had the approximate composition 23 pct Ni, 3.7 pct Mn, 0.05 pct C, 0.015 pct N, balance Fe, on a weight basis. This alloy is equivalent to about a 26 atomic pct Ni alloy, neglecting the carbon and nitrogen,[1] or about a 28 atomic pct Ni alloy including the carbon and nitrogen. It is close to the composition range for which isothermal nucleation should be observed, but not close enough according to Fig. 1. The fact that almost completely isothermal martensite was observed suggests that: 1—Fig. 1 is somewhat in error, or 2—the nickel-equivalent of manganese, carbon, and nitrogen has been underestimated, or 3—manganese alters the austenite/martensite interfacial free energy or the martensite shear angle. The third possibility is perhaps most likely, as the value of $(\theta^2\sigma^3)$ deduced for Cech and Hollomon's alloy[1] was about six times larger than that in Eq. 4 deduced for Fe-Ni alloys.

The nucleation C-curves for nickel contents less than about 0.29 Ni suggest that, experimentally, a temperature very similar to an M_s should exist for these alloys. The nucleation rate changes from a negligibly small value to a very large one in a few degrees temperature drop. The temperature at which martensite would first be observed, then, in any experiment where the specimen temperature is steadily lowered, would be that for which the martensite nucleation rate became appreciable, say,

$$\dot{n} = 1 \text{ nucleus per cc per sec} \qquad [5]$$

about 0.001 of the maximum rate observed by Cech and Hollomon (the results are very insensitive to the actual value selected for $\dot{n}$, as long as it is within a factor of a thousand or so of 1).

Taking Eq. 5 as the M_s criterion and taking N_V in Eq. 1 to be about 10^{26}, the M_s criterion becomes

$$W^*/kT = 82.9$$

or

$$\Delta F'T = 6.55\ (10)^{-11}\ (\theta^2\sigma^3) \qquad [6]$$

for ΔF in cal per mol. Taking the temperature for 10 pct transformation on continuous cooling at 10°C per min as the M_s temperature for Fe-Ni alloys, and using the data of Jones and Pumphrey,† M_s temperatures are fairly well defined by the relationship

$$\Delta F'T = 6.5\ (10)^{11} \qquad [7]$$

The agreement is shown in Fig. 2. The value of $(\theta^2\sigma^3)$ required is that given in Eq. 4.

Also shown in Fig. 2 is the curve for $\Delta F = 0$, which defines the temperature at which austenite and martensite are in metastable equilibrium. It is interesting to note that transformation in the temperature range $1000°K \leq T \leq 1180°K$ follows the curve $\Delta F = 0$, whereas transformation below 700°K follows Eq. 7.

The fact that transformation at high temperatures occurs at approximately the temperature where austenite and martensite are in equilibrium indicates one of two things: Either, 1—there is no appreciable nucleation barrier and no composition change during transformation, or 2—there is an appreciable nucleation barrier and a compensating composition change, giving a false significance to the curve $\Delta F = 0$. Assuming that the agreement with the curve $\Delta F = 0$ is not fortuitous, it appears that high temperature transformation is not impeded by strain energy,

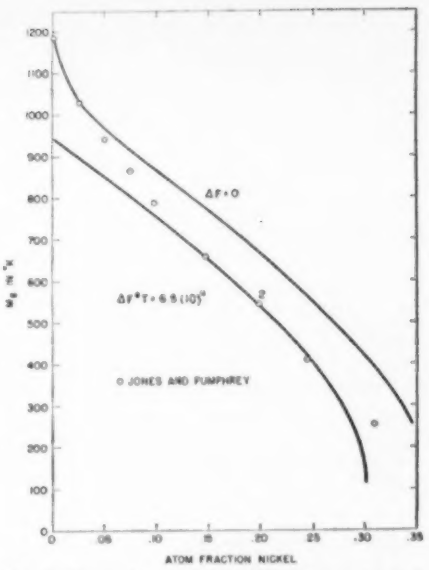

Fig. 2—Comparison of calculated and experimental temperatures for beginning of transformation in continuously cooled Fe-Ni alloys of varying nickel content. Experimental data taken from Jones and Pumphrey.[3]

either during nucleation or during growth. According to this view, the strain energy that normally accompanies martensite nucleation and growth is continuously relaxed at high temperatures. It might be, for example, that transformation proceeds by nucleation of ferrite at grain boundaries and edges, where stress relaxation and favorable interfacial free energy changes will allow easy nucleation, followed by noncoherent growth without composition change. In any event, whatever the interpretation at high temperatures, martensite appears to form in its characteristic fashion below 700°K.

Experimentally, the course of transformation on quenching an Fe-Ni alloy and holding it at constant temperature is as follows: In the first few seconds, while the specimen is coming to temperature, a considerable fraction of the volume is transformed to martensite. Subsequently, isothermal transformation is observed at a decreasing rate. At first glance it is not clear how the proposed isothermal nucleation description can apply to observations of this type. A clue to the answer lies in the fact that a constant rate of nucleation corresponds to an ever decreasing volume rate of transformation, owing to partition of the austenite by previously formed martensite plates. New plates are confined to smaller austenite volumes, and transform less material.

Two sorts of calculations can be made to check the idea that partitioning of the austenite can lead to the observed volume transformation effects. The first is a calculation of the volume transformed as a function of temperature for cooling at a constant rate, these data having been measured by Jones and Pumphrey.[3] The nucleation rate is known as a function of temperature from Eq. 1 or Fig. 1, and the volume fraction transformed depends upon the total number of nuclei according to the relationship[1]

$$V = 1 - [1 + (1 + f)\ n/n_v]^{-f/(1+f)} \qquad [8]$$

where V is the volume fraction transformed; n, the number of nuclei; n_v, the number of austenite grains per cc; and f, the fraction of the austenite trans-

† The M_s temperature had to be placed at 10 pct transformation because Jones and Pumphrey reported temperatures for 10, 50, and 90 pct transformation only.

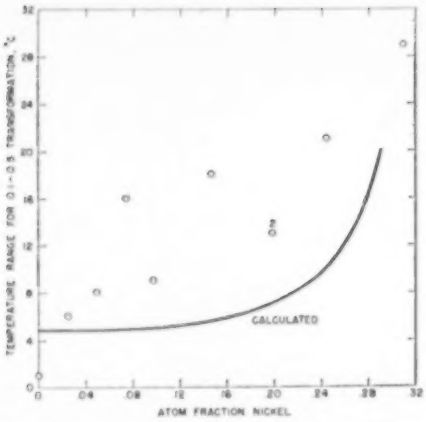

Fig. 3—Comparison of calculated and experimental widths of temperature ranges for 10 to 50 pct transformation on continuous cooling of Fe-Ni alloys. Data from Jones and Pumphrey.[3]

formed to martensite in the local volume where a typical plate forms. Taking $f \approx 0.05$, as suggested by analysis of Cech and Hollomon's data,[2] and n_v as 10^5 grains per cc, the width of the temperature range for 10 to 50 pct transformation at a cooling rate of 10°C per min was calculated as a function of nickel content. It is compared in Fig. 3 with the range as determined by Jones and Pumphrey. The calculation cannot be extended to 90 pct transformation, for which experimental data are available, because Eq. 8 breaks down above about 50 pct transformation. There is agreement to within a factor of two, which may be considered as reasonable.

The second calculation of interest in clarifying the role played by partition in converting a constant nucleation rate into a sharply varying transformation rate is that of determining the fraction transformed as a function of time for a typical alloy. Selecting a 29 pct Ni alloy and temperatures in the neighborhood of 200°K, the calculated fraction transformed is plotted vs time in Fig. 4. The behavior illustrated in Fig. 4 is very similar to that reported by Das Gupta and Lement[6] for the transformation of a high chromium steel containing about 15 pct Cr and 0.75 pct C, and by Kulin and Speich[7] for a 14 pct Cr, 9 pct Ni steel. The results for both alloys were

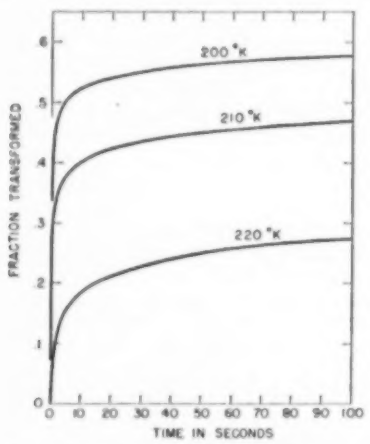

Fig. 4—Calculated isothermal transformation curves for an Fe-29 pct Ni alloy at three temperatures.

described in terms of two classes of martensite: that formed on cooling, and that formed isothermally. Actually, as suggested by Fig. 4, the martensite formed on cooling may have done so by thermal nucleation, in the same way as the subsequent isothermal martensite.

It should be pointed out that martensite that forms by rapid thermal nucleation, even though it forms during cooling, is not athermal martensite of the type described for steels containing significant amounts of carbon.[5] In a carbon steel, the *number* of active nuclei is determined by frozen-in fluctuations of carbon concentration, and is a function only of the temperature. For Fe-Ni alloys, on the other hand, it is suggested here that there is no characteristic number of nuclei. Rather, there is a characteristic *rate* of thermal nucleation for each temperature, the rate increasing so rapidly with decreasing temperature that a fairly sharp "beginning of transformation" temperature is observed on continuous cooling. Experimentally, an isothermal run in the neighborhood of the M_s temperature should give an essentially constant number of nuclei in a carbon steel, and an essentially constant nucleation rate in a substitutional iron alloy.

It can be concluded that a kinetic description of martensite formation in Fe-Ni alloys, and therefore very probably for other substitutional iron alloys, can be made in terms of homogeneous thermal nucleation. This description is in agreement with the following experiments: 1—Completely isothermal transformation in alloys of a very limited composition range; 2—Absence of transformation in more highly alloyed austenite; 3—M_s temperature as it depends upon composition in less highly alloyed austenite; 4—Temperature range for transformation on continuous cooling; and 5—Subsequent isothermal transformation in alloys where a considerable volume fraction transforms during cooling.

A more accurate treatment of the martensite transformation in substitutional iron alloys would require that attention be given to its autocatalytic nature.[9] Since the rate of thermal nucleation is increased by shear and tensile stresses, and in particular by stresses in the neighborhood of previously formed martensite plates, prior transformation can markedly increase the nucleation rate.[10] Nevertheless, it appears that the characteristic features of the martensite transformation in substitutional iron alloys are the result of thermal nucleation that, as a first approximation, can be considered homogeneous.

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Calculation of Martensite Nucleus Energy Using The Reaction-Path Model

by J. C. Fisher and D. Turnbull

ACCORDING to the "reaction-path" model[1,2] of martensite nucleation, the shear angle of the embryonic martensite plate must be treated as a variable, and included in any calculation of nucleus critical size. Also, as can be deduced from this model, the interfacial free energy between austenite and martensite does not reach its final value until the shear is completed. It is zero for zero shear angle. However, in order to account for the kinetics of the martensite transformation, some sort of interfacial energy barrier appears to be necessary even with the reaction-path model, for otherwise the volume and the energy of formation of the critical size nucleus both collapse to zero.[3] Cohen independently suggested that surface energy could be incorporated into the reaction-path model, with the overall free energy of a martensite embryo being a function of its volume and shear angle.[4]

It is possible to estimate the energy associated with the formation of a critical-size martensite nucleus starting with the reaction-path model and including a surface free-energy barrier. As the dependence of interfacial free energy upon shear angle is unknown, a simple type of dependence will be assumed, with the belief that the true dependence would not lead to appreciably different results.

Consider the work required to form a lenticular martensite plate with radius r, thickness t, and shear angle θ. There are three contributions; one being the interfacial free energy, one being the free energy change in the martensite plate, and one being the free energy increase in the surrounding austenite.

The interfacial free energy σ is assumed to depend upon the shear angle θ according to the relationship

$$\sigma = \sigma_e(\theta/\theta_e)^n \qquad [1]$$

where θ_e is the equilibrium shear angle and n is an exponent that may lie in the range $0 \leqq n \leqq 2$. The work required to form the interfaces of a martensite plate then is

$$W_s = 2\pi r^2 \sigma_e(\theta/\theta_e)^n \qquad [2]$$

The free energy change per unit volume of martensite is composed of two parts, one the ordinary volume free energy Δf_v which is negative, and the other the elastic strain energy $G\gamma_m^2/2$, where G is the shear modulus and γ_m the shear strain relative to the martensite structure. This expression for the strain energy is valid only when the shear strain γ_m is sufficiently small that the martensite is within its linear elastic range. There is no doubt that γ_m lies beyond the linear elastic range for embryos that are considerably subcritical. However, for critical nuclei it will be shown that γ_m is 1.5 pct or less, within the linear elastic range of martensite. For embryos of nearly critical size, then, the strain energy of the martensite is correctly given by $G\gamma_m^2/2$. The shear strain in the martensite is $\gamma_m = \theta_e - \theta$, and the work required to form the strained martensite is

$$W_m = (\pi r^2 t/2)\,[\Delta f_v + G(\theta_e - \theta)^2/2] \qquad [3]$$

The free energy change in the austenite is entirely that due to elastic distortion. The elastic strain is not uniformly distributed in the austenite, being large near the martensite plate and small elsewhere. Approximately, however, the energy corresponds to a uniform shear strain

$$\gamma_a = (\theta t/2)/r \qquad [4]$$

throughout the volume $4\pi r^3/3$ surrounding the plate. The work required to strain the surrounding austenite then is

$$W_a = (4\pi r^3/3)\,(G\gamma_a^2/2) = (G\theta^2/6)\,\pi r t^2 \qquad [5]$$

For simplicity, the same shear modulus G is assumed for each structure.

The total free energy for forming a plate then is

$$W = W_s + W_m + W_a = 2\pi r^2 \sigma_e(\theta/\theta_e)^n + (\pi r^2 t/2)\,[\Delta f_v + G(\theta_e - \theta)^2/2] + (G\theta^2/6)\,\pi r t^2 \qquad [6]$$

This expression is correct for nuclei and for embryos of nearly critical size, where, as will be shown, the strain energy in the martensite is correctly given by the expression $G(\theta_e - \theta)^2/2$. Having W as a function of r, t, and θ, as in Eq. 6, there is a saddle-point where W has a stationary value, W subsequently decreasing indefinitely as the nucleus volume increases along the reaction path. The stationary value of W is the energy of the critical nucleus.

The critical nucleus has radius, thickness, and shear angle such that $\partial W/\partial r = \partial W/\partial t = \partial W/\partial\theta = 0$. Performing these differentiations and calculating the critical nucleus energy,

$$W^* = [8192\pi(G\theta_e^2/6)^2\sigma_e^3/27\,\Delta f_v^4]\,\alpha \qquad [7]$$

where

$$\alpha = (\theta/\theta_e)^{3n+4}/[1 + G(\theta_e - \theta)^2/2\,\Delta f_v]^4 \qquad [7a]$$

and where θ is to be determined from the equation

$$(1 + 3n/4) + G\theta(\theta_e - \theta)/[\Delta f_v + G(\theta_e - \theta)^2/2] = 0 \qquad [8]$$

For Δf_v near -200 cal per mol or -10^9 ergs per cc, and θ_e near 1/6, as for iron-base alloys, Eq. 8 gives

$$\theta_e - \theta \approx -(4 + 3n)\,\Delta f_v/4G\theta_e \qquad [9]$$

as the difference between the equilibrium shear angle and the actual shear angle for a critical nu-

Table I. Values of $\theta_e - \theta$, θ/θ_e, and α as Functions of n

n	$\theta_e - \theta$	θ/θ_e	α
0	0.0060	0.964	0.93
1	0.0105	0.937	0.80
2	0.0150	0.910	0.63

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Discussion on this paper, TP 3533E, may be sent, 2 copies, to AIME by Dec. 1, 1953. Manuscript, Dec. 5, 1952. Cleveland Meeting, October 1953.

cleus. Even for n as large as 2, the difference does not exceed $\theta_o - \theta \simeq 0.015$, an elastic shear strain in the martensite of about 1.5 pct. This strain is within the linear elastic range of martensite, as tentatively assumed above.

Values for $\theta_o - \theta$ and θ/θ_o can be calculated from Eq. 9 for various values of n. Throughout the calculation it is assumed that $\Delta f_v = -10^9$ ergs per cc, $G = 10^{12}$ dynes per sq cm, and $\theta_o = 1/6$. The results are given in Table I.

Using the tabulated values of $\theta_o - \theta$ and θ/θ_o, the value of α in Eqs. 7 and 7a can be determined. The result is given in the final column of the table. It is evident that α is relatively insensitive to n, and is nearly unity. The quantity α is even more insensitive to Δf_v, increasing by 3 pct for $n = 0$ and by 10 pct for $n = 2$ when Δf_v is halved. In contrast, the term Δf_v^4 in Eq. 7 changes by 1600 pct at the same time, so that almost the entire temperature dependence of W^* lies in the bracketed coefficient of α.

In view of the above discussion, and of the other approximations involved in the nucleation calculation, α can be replaced by unity in Eq. 7 with little loss in rigor. Making this substitution,

$$W^* = 8192\pi(G\theta_o^3/6)\,\sigma_o^3/27\,\Delta f_v^4 \qquad [10]$$

Eq. 10 is the expression derived earlier by Fisher, Hollomon, and Turnbull[5] for martensite nucleation. It has met with success in describing the athermal nucleation of martensite in Fe-C alloys, and the thermal nucleation of martensite in Fe-Ni-Mn[6] and Fe-Ni[7] alloys.

It now appears that the reaction-path model, with a surface free-energy barrier introduced, gives essentially the same magnitude for the critical frequency of martensite nucleus formation as the nucleation theory of Fisher, Hollomon, and Turnbull. According to either view, the nucleation of martensite requires that a free-energy barrier be surmounted. Its height, according to either view, is given by Eq. 10.

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Technical Note

Effect of Nickel and Molybdenum on Stabilization Of the Austenite-Martensite Transformation

by D. J. Blickwede

PRESENT knowledge of the effects of time and temperature upon stabilization of the austenite-martensite reaction in steel is fairly complete,[1-3] but at the time the work described herein was initiated there was no information about the effects of alloy elements on the phenomenon. However, it has been assumed on theoretical grounds that alloy elements, particularly carbide formers, may have a significant effect on stabilization.[4] The present work was undertaken to provide information about the effect of two common elements, nickel and molybdenum, on stabilization in hypereutectoid and hypoeutectoid steels.

Seven induction-furnace melted, 1.2 pct C steels with a variation of nickel from 0 to 6 pct and of molybdenum from 0 to 1.0 pct were used for most of the experiments. Along with these a series of 0.4 pct C steels containing 0 to 6 pct Ni were also studied. The chemical compositions of the steels are listed in Table I. Stabilization in these forged, hot-rolled, annealed, and centerless ground steels was measured by aging brine-quenched specimens (austenite temperature, 1800°F) at 95° and 150°F and then determining the change in specific volume produced by subsequent cooling from room temperature to −320°F.[5]

In presenting the results the decrease in the change in specific volume, $\delta\Delta V$, after various stabilization times is used. This is given by

$$\delta\Delta V = \Delta V_o - \Delta V_t$$

where ΔV_o is the change in specific volume on quenching directly to −320°F and ΔV_t is the change in specific volume on quenching to −320°F after stabilizing for time t. In other words, $\delta\Delta V$ is a direct measure of the amount of martensite that is lost to the subzero quench as a result of stabilization and hence is greater, the longer the aging time or the higher the aging temperature.

The results are shown in Fig. 1. The scatter of the data, which are not shown for clarity and brevity, was sufficient to make the relative location of the curves on the $\delta\Delta V$ axis inaccurate within $\pm 2\times10^{-5}$ cc per g. However, some generalities appear which are of interest.

Effect of Nickel

In the 0.4 pct C steels, up to 3 pct Ni has little, if any, effect on the rate or amount of austenite stabilized at 95°F, Fig. 1a. Increasing nickel from 3 to 6 pct almost doubles the amount of austenite stabilized in a given time.

In the 1.2 pct C steels aged at 95°F, increasing the nickel from 1 to 3 pct appears to decrease the amount of stabilization, and a further increase to 6 pct Ni completely eliminates stabilization as shown in Fig. 1b (M_s of 6 pct Ni steel was below room temperature). The same behavior was exhibited by these steels stabilized at 150°F, especially after aging for periods longer than 5 hr, Fig. 1c.

Thus it appears that up to 1 pct Ni has little effect on stabilization at low or high carbon levels. More than 1 pct Ni increases stabilization at low carbon contents and decreases it at high carbon contents. Ward, Jepson, and Rait[6] have found that at inter-

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TN 167E. Manuscript, Jan. 17. 1953; revision, April 24, 1953.

Table I. Composition of Steels

Steel No.	C	Mn	P	S	Si	Ni	Mo
1	0.40	0.33	0.030	0.013	0.30	0.03	—
2	0.40	0.33	0.030	0.014	0.34	0.50	—
3	0.41	0.30	0.032	0.013	0.32	1.02	—
4	0.40	0.33	0.030	0.013	0.32	2.99	—
5	0.40	0.32	0.026	0.014	0.32	6.02	—
6	1.23	0.35	0.018	0.023	0.28	—	—
7	1.16	0.34	0.015	0.024	0.40	0.50	—
8	1.20	0.35	0.010	0.019	0.33	1.03	—
9	1.22	0.28	0.015	0.020	0.32	3.20	—
10	1.22	0.29	0.015	0.021	0.42	6.02	—
11	1.22	0.35	0.021	0.022	0.36	—	0.28
12	1.25	0.33	0.024	0.026	0.34	—	1.00

mediate carbon contents (0.9 to 1.0 pct C) increasing nickel decreases the amount of austenite stabilized in a given time at 75°F.

It is possible to explain these diverging effects of nickel in high and low carbon steels on the basis of the reaction-path theory for the austenite-martensite transformation.[2, 3] According to this theory nucleation of the martensite plate is considered to start in a strain embryo that comprises a state somewhere between austenite and martensite. The stabilization phenomenon is believed to be the relaxation of these strain centers. Since a large fraction of them are supplied by prior martensite formation, their number may be considered roughly proportional to the amount of martensite in the steel. Thus the number

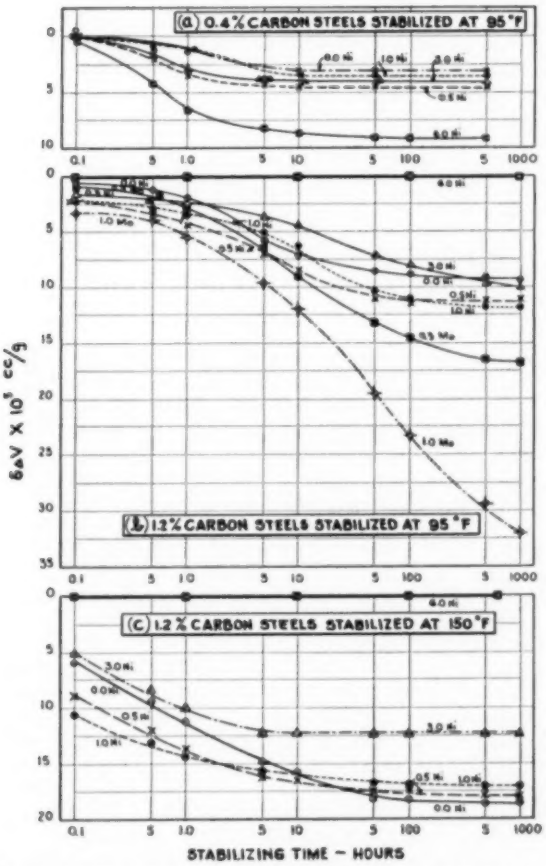
(a) 0.4% CARBON STEELS STABILIZED AT 95°F
(b) 1.2% CARBON STEELS STABILIZED AT 95°F
(c) 1.2% CARBON STEELS STABILIZED AT 150°F
δΔV X 10⁵ cc/g
STABILIZING TIME — HOURS

Fig. 1—Specific volume change on quenching to —320°F that is lost as a result of aging ($\delta\Delta V = \Delta V_o - \Delta V_t$).

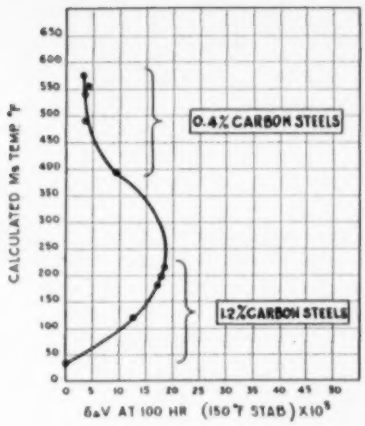

Fig. 2—M_s temperature vs decrease in change of specific volume ($\delta\Delta V$) due to stabilization of nickel steels at 150°F.

of strain centers capable of being stabilized would be larger, the greater the degree of undercooling below M_s. However, the amount of austenite available for stabilization becomes less as the steel is cooled further below M_s. The net result of these two opposing effects would cause stabilization at a given temperature to go through a maximum as the M_s temperature is decreased. This behavior seems to be exhibited by the low and high carbon nickel steels investigated here, as shown in Fig. 2. Thus in the 0.4 pct C steels the amount of austenite available for stabilization is low, but increases as the nickel content is raised; the result being an increase in stabilization. In the 1.2 pct C steels the number of strain embryos is low and decreases as the nickel content is raised, thereby decreasing stabilization. In other words, nickel appears to have little or no direct effect on stabilization, but influences the phenomenon indirectly through its effect on the amount of martensite in the as-quenched steel.

Ward, Jepson, and Rait[5] have found a similar behavior in a series of 1.0 pct C steels with increasing manganese from 0.3 to 6.0 pct. Evidently, at 1.0 pct C such a variation of manganese includes the maximum in the stabilization vs M_s temperature curve.

Effect of Molybdenum

The influence of molybdenum on stabilization of 1.2 pct C steels at 95°F is shown in Fig. 1b. In contrast to nickel, molybdenum markedly increases stabilization at this carbon level. This is in accord with published results for chromium[5] and theoretical considerations.[4]

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Further Progress in the Development of Mg-Zr Alloys to Give Good Creep and Fatigue Properties Between 500° and 650°F

by C. J. P. Ball, A. C. Jessup, P. A. Fisher,

D. J. Whitehead, and J. B. Wilson

The properties of a new magnesium alloy ZT1 containing 3.0 pct Th, 2.5 pct Zn, 0.7 pct Zr are described. The alloy possesses good creep and fatigue resistance up to 650°F, is free from microporosity, and is readily sand-cast. ZT1 is shown to be superior to zinc-free Mg-Th-Zr alloys.

THE advent of the jet engine created a demand for a light sand-casting alloy capable of being cast into large complex shapes and capable of resisting creep at temperatures up to 500°F. This demand has been largely met by alloys of the magnesium-cerium mischmetal-zirconium types, containing about 3 pct Ce mischmetal and 0.7 pct Zr, typified by Elektron magnesium alloys MCZ and ZRE1, the latter alloy also having a zinc addition of 2.5 pct. These alloys utilize the valuable properties at elevated temperatures conferred by the addition of rare earth metals to magnesium, a fact which has been known for many years and discussed by several investigators.[1-7] Murphy and Payne[8] showed that the addition of the powerful grain-refining element zirconium to Mg-Ce mischmetal alloys produced alloys with attractive room temperature properties combined with good creep properties and castability.

With the increasing power of modern jet engines the temperatures to which some of the magnesium parts are subject are likely to exceed the safe working range of the cerium-mischmetal-containing alloys, and at the beginning of the research considerable interest had been shown by aeroplane engine designers in a magnesium sand-casting alloy suitable for service at higher temperatures, e.g., 600°F and above. At an early stage in the development of this type of alloy a check on the fatigue properties at elevated temperatures indicated that, since the fatigue limit even at 10^8 reversals was appreciably higher than any permissible creep stress, the main emphasis of the research could be concentrated on creep behavior. The primary aim in developing the new alloy has therefore been to attain maximum creep resistance; but due consideration has also been given to other important properties such as castability, tensile strength, and cost.

It is by now well established that it is not possible to correlate creep strength with other short time mechanical properties such as are obtained in tensile

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Discussion on this paper, TP 3546E, may be sent, 2 copies to AIME by Dec. 1, 1953. Manuscript, Nov. 18, 1952; revision, April 24, 1953. Cleveland Meeting, October 1953.

tests.* Creep testing is therefore essential in developing an alloy for high temperature service. Furthermore experience shows that it is very dangerous to attempt to predict long term creep behavior on the basis of short term tests.

* Such apparent correlations, at least for magnesium-base alloys, originated at a time when the number of alloys was considerably smaller than at present and when little information was available on creep strength at elevated temperatures. Thus the theory that ductility at operating temperatures adversely influences the creep resistance of an alloy, once widely held and even now occasionally expressed,[9] may readily be proved false. For example, tensile tests at 400°F indicate that AZ91 alloy (in the as-cast condition) has an elongation of about 10 pct while Elektron ZRE1 has an elongation of about 30 pct. The creep resistance of ZRE1 is, however, well known to be many times better than that of AZ91 at 400°F.

The creep deformation of metals under constant load and at constant temperature is known to occur in three stages. In the first stage (primary creep) the rate of deformation is fairly rapid at the beginning, but soon decreases. In the second stage (secondary creep) the rate of deformation is constant. In the third stage (tertiary creep) the rate increases until the specimen fractures. The amount of deformation, and the time over which each stage persists, depends on the alloy, the temperature of the test, and the stress applied. For any given alloy an increase in temperature or stress usually results in increased deformation in the primary stage, an increase in the rate of deformation in the second stage, and a shorter period of secondary creep leading to earlier onset of tertiary creep and earlier fracture of the specimen. At very high temperatures or loads the secondary stage of creep may not be detectable, the specimen extending rapidly throughout the duration of test.

In developing a creep-resistant alloy the object is to obtain the minimum deformation in the first stage and the minimum rate of deformation, together with the maximum duration, in the second stage. It is very important to note that the onset of the third stage must be avoided in service since, at this point, fracture of the specimen may occur rapidly, the time being subject to wide and unpredictable variations. It is also very important to appreciate that the onset of the third stage of creep cannot be predicted without prior knowledge of the creep behavior of the material under examination. In other words, in examining a new material, the fact that a specimen has extended for a given time at a constant rate is no guarantee that it will continue to do so.

The evolution of a new high temperature alloy therefore necessitates the careful determination of its creep behavior over the whole range of its anticipated service life. Where this is not possible, for example in ground turbine installations designed for a life of 10 or 20 years, the only safe procedure is to use a sufficiently high safety factor which, however, almost inevitably means that the full properties of the alloy are not utilized. For aircraft the need to utilize material strength to the maximum, in order to avoid weight penalties, makes it imperative to examine the behavior of the material over the whole anticipated service life. Fortunately, the design life of aircraft engines is short enough to render such examination practicable. It is appreciated that where components are being designed for a short life—as in the case of certain engines for fighter aircraft—a choice of alloys may be made on the basis of 100 or 400 hr test results. In practice, creep results obtained from long term tests are perfectly satisfactory for short life evaluations; the converse unfortunately is not true.

In the development of this latest addition to the ultra light, high temperature alloys a service life of 1000 hr has been anticipated and the creep tests have been carried out for at least this time. It is probable that, when the full potentialities of the gas turbine are developed for aircraft use, a test of 1000 hr duration will prove to be much too short and 5000 or 10,000 hr tests may become necessary.

Evaluation of Creep Data

The present authors are strongly of the opinion that if creep data are not to be misleading they should be presented in a form which illustrates plainly the interdependence of stress, strain, and time for a given temperature. The utilization of the conventional "creep limit" derivation,[10, 11] does not fulfill the above requirement, and the information needed by the engineer is best presented as a family of curves relating stress to time at a given temperature for a series of values of strain, e.g., 0.001, 0.005, 0.01, and 0.05 pct. The maximum duration of time shown on the graph should be at least that expected as the maximum service life of components made from the material in question.

Where the time and available equipment have not been adequate for the completion of such a diagram, the present authors feel that in the absence of accurately drawn strain/time creep curves, full details of creep rates and strains should be given wherever possible. This method of presentation, although somewhat cumbrous to the reader, is the most informative and the least misleading, and has therefore been adopted in this paper.

During the work now recorded considerable thought was given to the possible utility of *"time to rupture"* tests, particularly in view of their wide use in the literature and for specification testing, particularly of turbine blade materials. A number of pilot tests convinced the authors that, owing to the large extension observed before rupture, the test was of very doubtful utility for the purpose of alloy development, since in every case component failure by extreme distortion would have preceded rupture. Provided that long term tests have been performed, there seems no reason why *"time to rupture"* tests should not be employed as acceptance tests whenever a satisfactory correlation with the longer tests can be obtained.

Previous Work on Mg-Th Alloys

The first indication that thorium has a beneficial effect on the creep resistance of magnesium was discovered by Sauerwald[12] who investigated both cast and wrought alloys. Leontis (whose work appears to have been carried out roughly in parallel with that described in the present paper) has published excellent surveys of the properties of Mg-Th and Mg-Th-Ce alloys[10] and the effect of zirconium on such alloys.[11] Leontis concluded that a Mg-3 pct Th-Zr alloy represented the optimum composition.

Experimental Procedure

Tensile creep tests were performed on three types of machine, namely: 1—5-ton Denison machines made to a design of the National Physical Laboratory and fitted with modified Martens extensometers, 2—direct loading machines of the British Non-Ferrous Metals Research Association type, and 3—miniature creep test units of the type described by Harris[13] and fitted with extensometers. The tests were carried out in accordance with British Standards Specifications 1686/1950 and 1687/1950.

The alloys examined in the present investigation were made by diluting Mg-Th hardener alloy with

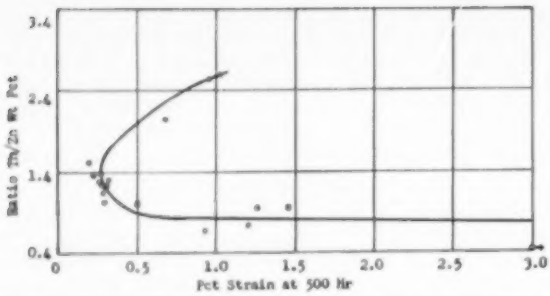

Fig. 1—Effect of Th-Zn ratio on creep resistance. Data plotted from results given in Table III.

pure magnesium and alloying with zirconium by reaction with a fused salt mixture (*"Master Salt"*) containing a reducible zirconium halide. Test pieces were in all cases machined from green sand-cast DTD bars.[14]

Thorium occurs with, and must be separated from, rare earth metals of the cerium group. The purity of the alloying materials used in this work was such that the rare earth metal content of the alloy did not exceed 3 pct of the thorium content. Rare earth metals were added when required in the form of commercial cerium mischmetal, an alloy of 45 to 55 pct Ce, the balance being other rare earth metals with a small amount of iron and silicon as impurities.

Because of the wide scatter generally expected in results obtained on creep tests, much consideration was given to the advisability of carrying out duplicate experiments. In view of the time involved it was decided not to duplicate each test but rather to carry out further tests at the same temperature and different stresses since, in addition to providing more information, the departure of the results obtained from a smooth curve would give an adequate indication of the scatter within the results. In fact, experience has indicated that the scatter obtained on the alloys developed during the present work is remarkably small compared with that shown by many other alloys with which we have worked. In the judgment of the authors, the values quoted are suitable for use in the design of high duty components.

Experimental Results

Mg-Th-Zr Alloys: Tensile tests on Mg-Th binary alloys showed that the room temperature mechanical properties were very poor, the ultimate tensile strength being of the order of 10,000 psi in the as-cast state for a 3 pct Th alloy. Addition of zirconium to give a full zirconium content of approximately 0.7 pct raised the tensile strength to approximately 22,000 psi, the yield strength to about 11,000 psi, and the elongation on 2 in. to about 5 pct in the as-cast condition. In view of the superior mechanical properties of the ternary alloys, creep tests were commenced on them. Table I gives the results of a survey of the ternary alloys with thorium content varying from about 2 to 5 pct.

These exploratory tests show that an alloy containing 3 pct Th develops tertiary creep at a stress of 1680 psi at 600°F in the as-cast state. The microstructure of this alloy consists essentially of solid solution with a thorium-rich intergranular network. This intergranular phase is capable of solution treatment at temperatures of the order of 1050°F, the solution treatment being accompanied by only slight grain coarsening. The results of creep tests on solution-treated material show that although the creep properties are markedly improved by solution heat treatment, the alloy still develops tertiary creep at stresses of 3360, and 1120 psi at 600° and 662°F, respectively.

Extension of the duration of solution treatment from 2 to 16 hr appears to be of no benefit to the creep resistance in the case of the alloy containing 3 pct Th.

The room temperature tensile properties of these alloys can be improved by a low temperature aging treatment following the solution treatment. The following properties were obtained with the alloy containing 3 pct Th and 0.7 pct Zr after solution-treatment for 2 hr at 1050°F followed by aging for 16 hr at 400°F, British DTD bars being used: 0.2 pct proof stress, 15,750 psi; ultimate tensile strength, 31,500 psi; and elongation on 2 in., 6 pct. Slightly lower tensile values are obtained if the aging treatment is carried out at 600°F, the following being typical results: 0.2 pct proof stress, 14,750 psi; ulti-

Table I. Results of Creep Tests on Sand-Cast Mg-Th-Zr Alloys

Analysis, Pct				Plastic Strain, Pct						
Th	Zr	Temperature, °F	Stress, Psi	100 Hr	300 Hr	500 Hr	700 Hr	1000 Hr	Creep Rate, In./In./Hr x10⁵	Initial Condition*
3.0	0.71	600	1680	0.07	0.18	0.4	0.8	1.6	300 (at 1000 hr)	As-cast
2.8	0.66	600	3360	0.55	9.0	21.8**			26,000 (at 350 hr)	As-cast
2.8	0.63	600	1680	0.045	0.12	0.33	0.465	0.68	71 (at 1000 hr)	Solution treated 2 hr at 1050°F
3.6	0.72	600	3360	0.3	1.1	3.75	9.2‡‡		4,400 (at 700 hr)	Solution treated 2hr at 1050°F
3.6	0.72	662	1120	0.08	0.30	0.60	1.03	1.78	280 (at 1000 hr)	Solution treated 2 hr at 1050°F
3.0	0.71	600	3360	0.36	6.7	16.6**			19,000 (at 350 hr)	Solution treated 16 hr at 1050°F
3.0	0.71	600	3360	0.38	4.6	17.4†			18,000 (at 380 hr)	Solution treated 2 hr at 1050°F plus 16 hr at 400°F
1.9	0.65	600	3360	1.38	>20††				10,000 (at 180 hr)	Solution treated 2 hr at 1050°F
4.7	0.66	600	3360	0.30	2.45	>24‡			19,000 (at 420 hr)	Solution treated 16 hr at 1050°F

* All specimens were held for 16 hr at the temperature of test before applying the load.
** At 360 hr. Fractured.
† At 384 hr. Fractured.
†† At 240 hr.
‡ At 450 hr.
‡‡ Fractured at 700 hr.
Note. The work described in this report was carried out in accordance with British practice with temperature measured in degrees centigrade and load in long tons (2240 lb). For convenience these figures have been calculated back to degrees Fahrenheit and pounds.

Table II. Results of Creep Tests on Sand-Cast Mg-Th-RE*-Zr Alloys at 1680 Psi at 600°F

Analysis, Pct			Plastic Strain, Pct					Creep Rate at 1000 Hr,	Initial
Th	RE*	Zr	100 Hr	300 Hr	500 Hr	700 Hr	1000 Hr	In./In./Hr x 10^7	Condition†
3.4	1.4	0.7	0.24	0.91	1.71	2.35	3.37	332	As-cast
3.0	3.3	0.68	0.195	0.4	0.57	0.73	0.98	85	As-cast
3.0	4.0	0.7	0.13	0.275	0.415	0.56	0.85	81	As-cast
3.0	3.0	0.68	0.13	0.385	0.74	0.975	1.36	124	Solution treated 2 hr at 1050°F

* RE is cerium mischmetal.
† All specimens were held for 16 hr at the temperature of test before applying the load.

mate tensile strength, 30,000 psi; and elongation on 2 in., 6 pct.

It appears however that the creep resistance at 600°F of the alloy containing 3 pct Th may be adversely affected by the use of an aging treatment at 400°F. Comparison of the results obtained with and without the treatment at 400°F indicates that a considerably higher creep rate was obtained when this precipitation treatment was included, Table I. The results in Table I also indicate that the optimum thorium content is probably of the order of 3 pct.

It is evident that, for the ternary alloy containing 3 pct Th in the solution-treated state, the maximum feasible working stress at 600°F for a creep rate of less than 5×10^{-6} in. per in. per hr at 1000 hr is approximately 1680 psi. The alloy suffers from the serious drawback that a high temperature solution heat treatment is essential for the development of optimum creep resistance. Protective atmospheres are required for the heat treatment of magnesium alloys at 1050°F and large complex castings would require careful jigging to avoid distortion.

Effect of Cerium Mischmetal Additions to Mg-Th-Zr Alloys: Results are given in Table II of an examination of the effect on creep resistance at 600°F of the addition of varying amounts of cerium mischmetal to the ternary Mg-Th-Zr alloy containing 3 pct Th.

The addition of up to 4 pct cerium mischmetal to this ternary alloy results in no improvement to the creep resistance obtainable in the solution-treated condition. Solution treatment of the ternary alloy to which 3 pct of cerium mischmetal has been added has no marked effect on the creep behavior at 600°F.

Effect of Zinc Additions to Mg-Th-Zr Alloys: Table III gives the results of creep tests carried out at a common stress of 3360 psi at 600°F on 20 compositions in the Mg-Th-Zn-Zr series.

The results are given in order of increasing Th/Zn ratio and plotted in Fig. 1. It can be seen that, within the limits investigated, there is a clearly defined relationship between creep resistance (as defined by the strain at 500 hr) and composition. The optimum Th/Zn ratio for best creep resistance occurs at about 1.3, corresponding with a nominal composition of 3 pct Th, 2.5 pct Zn.† This composition has been

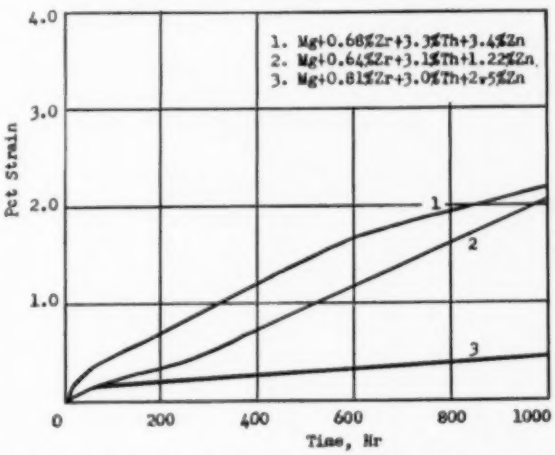

Fig. 2—Comparison of three alloys of equal thorium content with varying Th-Zn ratios. Tested at 3360 lb per sq in. at 600°F.

† From the curve plotted in Fig. 2, based on the results obtained to date, the following limits appear suitable for specification purposes: Th, 2.5 to 3.5 pct and Zn, 1.75 to 2.5 pct.

Table III. Results of Creep Tests on Sand-Cast Mg-Th-Zn-Zr Alloys at 3360 Psi at 600°F*

Analysis, Pct				Plastic Strain, Pct					Creep Rate at 1000 Hr,
Th	Zn	Zr	Th/Zn	100 Hr	300 Hr	500 Hr	700 Hr	1000 Hr	In./In./Hr x 10^7
1.9	4.7	N.D.†	0.4	1.56	9 (fractured)				2800 (250 hr)
2.6	3.9	N.D.	0.67	0.45	0.77	0.92	1.10	1.50	75 (800 hr)
2.0	2.7	0.65	0.75	3.0	0.68	1.2	2.1	—	430 (800 hr)
3.85	4.1	0.74	0.94	0.32	0.78	1.25	—	—	240 (500 hr)
3.3	3.4	0.68	0.96	0.45	0.92	1.45	1.82	2.16	95
2.7	2.7	0.76	1.0	0.19	0.33	0.49	0.65	0.89	82
2.8	2.65	0.67	1.05	0.21	0.34	0.45	0.62	0.88	88
2.6	2.5	0.77	1.04	0.17	0.32	0.50	0.67	0.91	88
2.5	2.4	N.D.	1.04	0.10	0.19	0.29	0.26	0.48	39
5.1	4.4	0.44	1.16	0.116	0.10	0.29	0.368	0.49	42
3.1	2.5	0.67	1.24	0.13	0.225	0.28	0.315	0.355	15
3.0	2.5	0.81	1.24	0.15	0.23	0.31	0.36	0.45	31
2.0	1.5	0.65	1.30	0.095	0.18	0.26	0.37	0.63	87
2.6	2.0	0.88	1.30	0.15	0.23	0.32	0.47	0.80	110
3.85	2.8	0.76	1.38	0.12	0.21	0.27	—	—	30 (500 hr)
3.10	2.0	0.75	1.55	0.12	0.15	0.19	0.22	0.28	20
3.1	1.5	0.66	2.06	0.24	0.46	0.67	0.89	1.21	100
3.1	1.22	0.64	2.54	0.19	0.53	0.95	1.38	2.05	230
3.65	1.4	0.67	2.60	0.33	0.71	1.02	1.16	1.28	45
1.9	0.7	0.70	2.7	0.2	0.6	0.90	—	—	190 (500 hr)

* The initial condition was as-cast. All specimens were held for 16 hr at the temperature of test before applying the load.
† N.D., not determined.

Table IV. Results of Creep Tests on Sand-Cast Mg-Th-Zn-Zr Alloys at 2240 Psi at 600°F*

Analysis, Pct				Plastic Strain, Pct					Creep Rate at 1000 Hr,
Th	Zn	Zr	Th/Zn	100 Hr	300 Hr	500 Hr	700 Hr	1000 Hr	In./In./Hr x 10^7
3.2	4.0	0.58	0.8	0.05	0.10	0.13	0.16	0.20	14
1.2	1.5	0.64	0.8	0.06	0.096	0.13	0.17	0.22	18
3.85	4.1	0.74	0.94	0.15	0.25	0.29	0.32	0.36	14
2.5	2.4	N.D.†	1.04	0.08	0.10	0.10	0.115	0.135	6
2.6	2.5	0.77	1.04	0.06	0.11	0.15	0.19	0.25	18
5.1	4.4	0.44	1.16	0.07	0.108	0.14	0.172	0.22	16
3.1	2.5	0.67	1.24	0.10	0.17	0.19	0.22	0.26	15
2.6	2.0	0.88	1.30	0.08	0.11	0.15	0.19	0.24	20
3.85	2.8	0.76	1.38	0.06	0.07	0.10	0.10	0.12	7
3.1	2.0	0.75	1.55	0.03	0.04	0.05	0.07	0.09	6
3.1	1.5	0.66	2.06	0.08	0.10	0.12	0.145	0.18	10
3.1	1.22	0.64	2.54	0.11	0.29	0.36	0.39	0.43	14
3.65	1.40	0.67	2.60	0.13	0.20	0.25	0.29	0.35	21

* The initial condition was as-cast. All specimens were held for 16 hr at temperatures of test before applying the load.
† N.D., not determined.

named Elektron magnesium alloy ZT1.‡ The marked effect of Th/Zn ratio on creep resistance is further shown by Fig. 2 which gives strain/time curves for

‡ The alloy composition (3.0 pct Th, 2.5 pct Zn, 0.7 pct Zr) is protected by U.S. Patent 2,604,396.

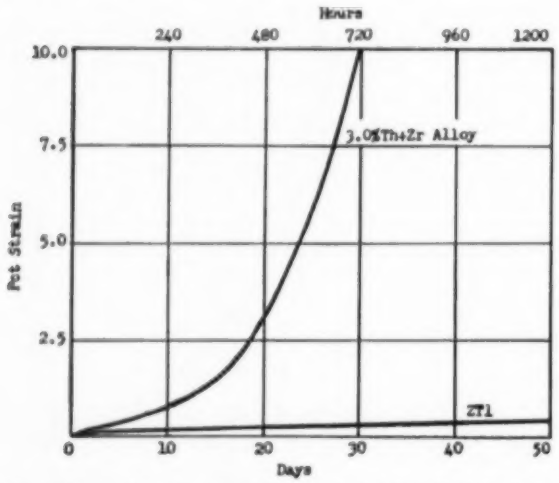

Fig. 3—Comparison of creep behavior of zinc-free thorium alloy with ZT1. Both alloys tested at 3360 lb per sq in. at 600°F.

tests at a stress of 3360 psi at 600°F on three alloys of sensibly equal thorium contents (3 pct) but with Th/Zn ratios of 0.96, 1.2, and 2.5. Tests on alloys containing 3.85 and 5.1 pct Th (Table III) indicate that under these conditions of test there is no advantage to be gained by increasing the thorium content over 3 pct while maintaining the Th/Zn ratio close to 1.3.

With thorium contents appreciably below 3 pct the results included in Table III indicate a tendency for the creep rate to increase toward the end of the 1000 hr test. It may be possible to produce an alloy having satisfactory creep resistance and containing as little as 2 pct Th, with the appropriate zinc content. It is evident, however, that the permissible variation in composition decreases as the thorium content is reduced and it remains for further investigation to determine whether an alloy containing as little as 2 pct Th can be safely employed in industrial practice. This point is now under investigation.

A number of tests on alloys of varying thorium and zinc contents have been carried out at a stress of 2240 psi at 600°F and the results are included in Table IV. It is evident that Th/Zn ratio is not so critical at the lower stress and a wide range of composition is capable of giving satisfactory creep rates. In fact all the alloys investigated, with Th/Zn ratios varying from 0.8 to 2.6, would be usable at a stress of 2240 psi at 600°F.

The very marked effect of zinc in improving the creep resistance of Mg-Th-Zr alloys is illustrated by Fig. 3 which shows the effect of a 2.5 pct Zn addition to a 3 pct Th, 0.7 pct Zr alloy when tested in creep at a stress of 3360 psi at 600°F. Although the zinc-free alloy is in the solution-treated condition it develops rapid tertiary creep after the 300 hr test, leading to ultimate fracture before the expiration of 1000 hr, whereas ZT1 alloy extends about 0.4 pct in 1000 hr.

Effect of Prior Heat Treatment on the Creep Resistance of ZT1: Results are given in Table V of creep tests carried out on ZT1 after heat treatment for 24 hr at temperatures from 750° to 930°F.

It is evident that the use of heat-treatment temperatures between about 700°F and about 930°F damages the creep resistance of ZT1. The solidus of this alloy is believed to be between 930° and 1025°F. The damaging effect on creep resistance apparently decreases as the solidus temperature is reached and this suggests that solution of a thorium-rich phase occurs at about 930°F. Below this temperature it is

Table V. Results of Creep Tests on ZT1 after Various Heat-Treatment Cycles. Tests at 3360 Psi at 600°F

Analysis, Pct			Plastic Strain, Pct					Creep Rate at 1000 Hr,	Initial
Th	Zn	Zr	100 Hr	300 Hr	500 Hr	700 Hr	1000 Hr	In./In./Hr x 10^7	Condition*
3.1	2.5	0.67	0.13	0.225	0.28	0.315	0.355	15	As-cast
3.3	2.6	0.78	0.22	0.39	0.49	0.60	1.01	180	H.T. 24 hr at 750°F
3.4	2.6	0.74	0.16	0.27	0.41	0.65	1.08	150	H.T. 24 hr at 880°F
3.4	2.6	0.74	0.22	0.35	0.47	0.59	0.76	58	H.T. 24 hr at 930°F

* All specimens were held for 16 hr at temperatures of test before applying the load.

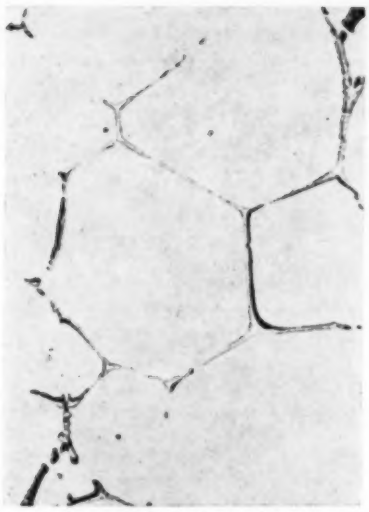

Fig. 4—Micrograph showing typical structure of Mg-Zn-Th-Zr alloy illustrating blue tortuous phase. X1000. Area reduced slightly for reproduction.

probable that precipitation of this phase occurs, the creep resistance being impaired by progressive coarsening of this precipitate as the heat-treatment temperature is increased, as suggested by Mellor and Ridley.[8] In this respect ZT1 alloy appears to behave in a manner similar to that of Elektron ZRE1.

Microstructural Features of Mg-Th-Zn-Zr Alloys: The microstructure of Mg-Th-Zn-Zr alloys is generally similar to that of the Mg-Ce-Zn-Zr alloys, the grain boundaries being well delineated by free intermetallic compound resulting from breakdown of the eutectic. Two distinct forms of grain boundary phase have been observed in Mg-Th-Zn-Zr alloys, one blue in color and tortuous, and the other brown and more acicular in shape. The type of grain boundary phase is dependent on the Th/Zn ratio. With high zinc contents, at ratios below about 0.5, only the blue tortuous phase is present, and with low zinc contents, at ratios above about 1.4, only the brown acicular phase is present.

The optimum creep resistance appears to be associated with compositions at which the brown phase preponderates over the blue. Reduction of zinc content below this point, although completing the conversion to the brown phase, results in a decrease in creep resistance. Typical micrographs of the phases are shown in Figs. 4 and 5.

Effect of Cerium-Mischmetal Additions to Mg-Th-Zn-Zr Alloys: Partial Replacement of the Thorium Content of ZT1 with Cerium Mischmetal: In view of the relatively high cost of thorium as an alloying constituent, tests were carried out to determine whether a proportion of the thorium content of ZT1 could be replaced by rare earth metals without impairment of the creep performance at 600°F.

The first two tests of Table VI show that although ZT1 alloy in which about 50 pct of the thorium has been replaced by cerium mischmetal behaves satisfactorily at a stress of 1680 psi at 600°F, the creep resistance at 3360 psi is seriously affected. It is evident that economies cannot be effected in this manner without some sacrifice of creep strength.

Fig. 5—Micrograph showing typical structure of Mg-Zn-Th-Zr alloy illustrating brown acicular phase. X1000. Area reduced slightly for reproduction.

Addition of Cerium Mischmetal to ZT1 Alloy: In view of the deleterious effect of cerium mischmetal on Mg-Th-Zn-Zr alloys, further tests were carried out to investigate the damaging effect in more detail. Table VI gives the results of tests on ZT1 alloy containing up to 1.2 pct Ce mischmetal at 3360 psi at 600°F. It is evident from these tests that a rare earth metal content of 0.2 pct or greater has an adverse effect on creep resistance. With a rare earth metal content of greater than 0.2 pct there is a tendency for the alloy to develop tertiary creep after 500 hr.

Properties of ZT1 Alloy

The essential properties required of a high temperature material have been summarized[15] as follows: 1—creep resistance, i.e., resistance to deformation under the influence of a steady applied stress; 2—fatigue strength, i.e., resistance to cracking under the influence of a cyclic stress; 3—resistance to corrosion by the surrounding atmosphere (including

Table VI. Results of Creep Tests on Sand-Cast Mg-Th-RE-Zn-Zr Alloys at 600°F

Analysis, Pct				Stress,	Plastic Strain, Pct					Creep Rate at 1000 Hr,
Th	RE	Zn	Zr	Psi	100 Hr	300 Hr	500 Hr	700 Hr	1000 Hr	In./In./Hr x 10^7
3.1	<0.1	2.5	0.7	1,680	0.05	0.125	0.15	0.175	0.20	12
1.1	1.8	2.3	0.79	1,680	0.12	0.19	0.22	0.25	0.30	11
3.1	<0.1	2.5	0.7	3,360	0.13	0.225	0.28	0.315	0.355	15
1.57	1.52	2.5	0.65	3,360	1.1	1.92				980(150 hr)
3.1	0.2	2.6	0.72	3,360	0.06	0.15	0.24	0.30	0.72	130
3.1	0.5	2.5	0.72	3,360	0.17	0.32	0.46	0.63	1.24	250
3.1	0.8	2.6	0.73	3,360	0.22	0.43	0.64	0.90	1.63	250
3.0	1.2	2.6	0.69	3,360	0.40	—	—	—	—	400(250 hr)

* The initial condition was as-cast. All specimens were held for 16 hr at the temperature of test before applying the load.

oxidation and scaling at operating temperature); 4—resistance to thermal shock, i.e., resistance to cracking under the influence of stresses developed by rapid changes of temperature. In considering the specific requirements of a magnesium-base casting alloy for elevated temperature service, the following additional factors may be included: 5—satisfactory strength and ductility at room temperature; 6—ability to withstand high stresses of short duration at high temperature without excessive permanent set; 7—ease of casting; and 8—other factors of importance, including cost, weldability, pressure tightness, density and ease of machining.

It is of interest to see how ZT1 alloy meets these requirements.

Creep Resistance: Table VII gives the creep properties of ZT1 at 600° and 662°F. These properties are

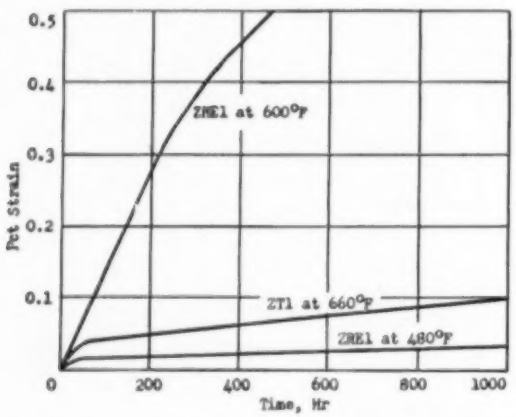

Fig. 6—Comparison of creep behavior of ZT1 and Elektron ZRE1 alloys. All tests at 1120 lb per sq in.

a considerable advance on the creep resistant magnesium alloys at present in use. For conditions of equal stress, ZT1 offers a permissible increase of about 120°F in service temperature over ZRE1 alloy. Fig. 6 illustrates the improved creep resistance of ZT1 as compared with ZRE1 alloy. Whereas ZRE1, although perfectly satisfactory at a stress of 1120 psi at 482°F, shows considerable deformation at 600°F, ZT1 is seen to display very satisfactory creep resistance at 1120 psi at 662°F.

Fatigue Strength: Table VIII gives the results of single point loading alternating bending fatigue tests on ZT1 alloy at room and elevated temperature.

Resistance to Corrosion and Oxidation: Corrosion tests on ZT1 alloy indicate that it has the same order of corrosion resistance as the existing high temperature magnesium alloys containing mischmetal, for example, a 28 day total immersion test in 3 pct salt solution saturated with $Mg(OH)_2$ gave for ZT1 (heat-treated 16 hr 600°F) a rate 1 mg per sq cm per day, and for ZRE1 (heat-treated 16 hr 350° to 400°F) a rate 1.5 to 2 mg per sq cm per day; and a 6 months sea water spray test gave for ZT1 (heat-treated 16 hr 600°F) a rate 0.4 mg per sq cm per day, and for ZRE1 (heat-treated 16 hr 350° to 400°F) a rate 0.3 to 0.5 mg per sq cm per day.

Although accurate determinations of the rate of oxidation at elevated temperatures have not been carried out on ZT1 alloy, it has been observed that exposure to normal atmosphere at temperatures up

Table VII. Creep Properties of ZT1 at Elevated Temperature

Temperature, °F	Stress, Psi	Plastic Strain, Pct					Creep Rate 1000 Hr, In./In./Hr x 10^7
		100 Hr	300 Hr	500 Hr	700 Hr	1000 Hr	
600	1680	0.10	0.125	0.147	0.170	0.20	12
600	2240	0.10	0.17	0.19	0.22	0.26	15
600	3360	0.13	0.225	0.28	0.315	0.355	15
662	1120	0.035	0.05	0.07	0.08	0.105	9
662	1680	0.09	0.25	0.475	0.90	1.99	35
662	2240	0.12	0.30	0.60	1.15	2.59	430

to 662°F for periods of up to 1000 hr results in only superficial tarnishing. No serious exfoliation has been observed at temperatures up to 752°F.

Resistance to Thermal Shock: The ductility of ZT1 alloy at room and elevated temperatures is more than adequate to insure freedom from cracking under stresses developed by rapid changes in temperature.

Mechanical Properties at Room Temperature: These are of importance in view of the fact that the castings must be capable of being machined and fitted without danger of cracking or distortion. DTD type test bars in ZT1 alloy, stabilized 16 hr at 600°F and tested at room temperature, give the following properties: 0.2 pct proof stress, 12,500 to 15,000 psi; ultimate tensile strength, 27,000 to 31,000 psi; and elongation on 2 in., 5 to 10 pct. These properties are a considerable advance on those obtainable from the rare earth metal containing, creep resistant Mg-Zr alloys at present in use, particularly as regards ultimate tensile strength. The room temperature tensile properties of ZT1 may therefore be considered as adequate.

Tensile Properties at Elevated Temperature: Table IX lists the high temperature tensile properties of ZT1. It can be seen that ZT1 shows good retention of tensile properties over the temperature range 527° to 662°F. In particular, it is to be noted that the value for limit of proportionality shows no drop over this temperature range. The progressive reduction in values for elongation with increase in the temperature of test is an unusual feature in a magnesium casting alloy. The tensile elongation appears to reach a minimum value of about 35 pct over a temperature range of 650° to 700°F but above this range the elongation increases progressively, and at 750°F is about 40 pct. The values for elongation are, however, well above any minimum likely to arise as a service requirement and this progressive decrease in elongation is therefore more of a metallurgical novelty than of practical significance.

Table VIII. Results of Fatigue Tests on ZT1 Alloy

Temperature of Test, °F	Fatigue Limit for $5x10^7$ Reversals in Psi. Notched*	Fatigue Limit for Specified Reversals in Psi. Unnotched		
		10^7	$5x10^7$	10^8
68	± 10,080		± 10,080	
392		± 7,170		± 7,170
482		± 6,050		± 6,050
600		± 4,700		± 3,360
662		± 3,470		± 2,350

* The notch used in this test had a stress concentration factor of 2.

Table IX. Elevated Temperature Tensile Properties of ZT1

Temperature of Test, °F	Limit of Proportionality, Psi	0.1 Pct Proof Stress, Psi	0.2 Pct Proof Stress, Psi	Ultimate Tensile Strength, Psi	Elongation, Pct on 2 In.
527	3300	7050	7900	12,100	50
572	3300	6800	7600	10,750	45
617	3300	6300	7100	10,080	40
662	3300	6100	6900	9,630	35

Castability of ZT1 Alloy: Several large castings of up to 200 lb in weight have been made in ZT1 alloy. The alloy is completely free from microporosity and can be handled in the foundry in the same manner as Elektron alloys ZRE1 and MCZ. A tendency to the formation of folds due to casting skins in thin sections has been observed in certain castings, but this can be overcome by the choice of suitable running techniques.

Other Factors: ZT1 alloy, being a magnesium alloy not subject to microporosity, can be cast into large thin walled shapes with the certainty that the resultant casting will be pressure tight provided it is free from cracks and hot tears. The casting characteristics of ZT1 are such that no trouble should be experienced with cracks or hot tears. Only a simple low temperature heat treatment is required.

Like Elektron magnesium alloys ZRE1 and MCZ, ZT1 is readily weldable using the techniques at present in use for magnesium alloys. This is an important factor in foundry economics and obviates the scrapping of castings for minor defects amenable to repair by welding.

ZT1 alloy possesses the same ease of machining as other magnesium alloys.

High Temperature Tensile Properties of Zinc-Free 3 pct Th Alloy

It was thought to be of interest to examine the high temperature tensile properties of the zinc-free 3 pct Th-Zr alloy under the same testing conditions as used for ZT1, and Table X gives the values obtained.

The two different temperatures for precipitation treatment were examined in order to provide a comparison with the values quoted by Leontis,[11] who used a precipitation temperature of 400°F, and to examine the effect of precipitation at 600°F in view of the apparent loss in creep strength when the lower temperature was used, see Table I.

The higher precipitation temperature results, as might be expected, in a decrease in values for limit of proportionality and proof stress but does not affect the value for tensile strength appreciably. The value for proof stress at 572°F may be compared roughly with that quoted by Leontis at 600°F and is about 2000 psi lower than he obtained. This is very probably due to the use of a test bar of different type§ and possibly to differences in method of testing.

Table X. Elevated Temperature Tensile Properties of 3 Pct Th, 0.7 Pct Zr Alloy

Temperature of Test, °F	Limit of Proportionality, Psi	0.1 Pct Proof Stress, Psi	0.2 Pct Proof Stress, Psi	Ultimate Tensile Strength, Psi	Elongation, Pct on 2 In.
Heat-Treated 2 Hr 1050°F, Plus 16 Hr 400°F					
572	5600	9400	10,700	17,500	24
662	2800	5500	6,400	13,200	30
Heat-Treated 2 Hr 1050°F, Plus 16 Hr 600°F					
572	4500	8000	9,200	17,000	18
662	2500	4900	5,800	15,000	25

§ Leontis appears to have used unmachined cast to shape bars, which give values for proof stress appreciably higher than those obtained from the British DTD bar.

In comparison with ZT1 it can be seen that the tensile properties of the 3 pct Th-0.7 pct Zr alloy are higher at 572°F but show a marked drop over the temperature range 572° to 662°F. The values for limit of proportionality and proof stress thus become lower than those for ZT1 at 662°F, the value for ultimate strength remaining above that for ZT1.

Conclusions

1—Mg-Th-Zn-Zr alloys are capable of showing a high level of creep resistance at temperatures of 600°F and above.

2—For optimum creep resistance, the zinc content must be closely controlled in relationship to the thorium content. A preferred composition of 3 pct Th, 2.5 pct Zn, 0.7 pct Zr has been determined: the composition has been termed ZT1.

3—ZT1 alloy possesses superior creep resistance to the equivalent zinc-free alloy.

4—The alloy possesses good fatigue properties at room and elevated temperatures combined with good tensile properties and corrosion resistance.

5—ZT1 alloy shows good retention of high temperature tensile properties over the range 527° to 662°F. In particular the value for the limit of proportionality shows no drop over this temperature range.

6—ZT1 alloy can be successfully cast in sand molds. The alloy is nonmicroporous and shows very good freedom from tendency to hot cracking.

7—Additions of cerium mischmetal are detrimental to the creep resistance of ZT1.

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Textures of Rolled and Annealed Iodide Zirconium

by J. H. Keeler, W. R. Hibbard, Jr., and B. F. Decker

Textures of hot-rolled, of cold-rolled, and of cold-rolled and annealed zirconium sheets were determined by use of an X-ray spectrogoniometer. All textures showed a tilt of the basal planes ±40° from the rolling plane about the rolling direction. Deformation textures showed the [10$\bar{1}$0] direction to be approximately in the rolling direction. After recrystallization, the [11$\bar{2}$0] direction was approximately in the rolling direction. This texture was sharpened by annealing above the allotropic transformation. Textures of sheet produced by alternately cold rolling and then annealing were less strongly oriented than sheets that underwent severe deformation without intermediate anneals.

THIS study of the deformation and annealing textures of high purity zirconium was carried out to supplement an investigation of zirconium and zirconium-base alloys. The spectrogoniometer method of pole figure determination was used and yielded more detailed and precise information than the photographic techniques used by previous investigators.

Earlier investigations of the rolling textures of hexagonal metals titanium,[1-3] beryllium,[3] and zirconium[4-6] having a c/a ratio slightly less than 1.6 disclosed a rotation of the basal planes from the rolling plane about the rolling direction so that the basal poles were inclined toward the transverse direction, and with one exception,[4] the [10$\bar{1}$0] direction was found parallel to the rolling direction. Similarly, hot rolling produced a spread of the basal poles in the transverse direction,[2,3,5] with the [10$\bar{1}$0] direction parallel to the rolling direction. The textures of these metals after recrystallization differ somewhat from each other although still exhibiting the transverse rotation of the basal poles about the rolling direction. Zirconium[6] was found to have the [11$\bar{2}$0] direction in the rolling direction, beryllium[3] continued to have the [10$\bar{1}$0] direction in the rolling direction, and titanium[2] was shown to have the [10$\bar{1}$0] direction in the rolling direction (the cold-rolling texture) when annealed at 1000°F, changing to the [11$\bar{2}$0] direction in the rolling direction when annealed at 1500°F. It was suggested[2] that the titanium retains the deformation texture after recrystallization, and preferred grain growth at higher temperatures produces the second texture. Annealing above the allotropic transformation temperature did not appreciably change the high temperature recrystallization texture for titanium.[2] Coarse-grained zirconium showed more spread[6] in the transformation texture than in the recrystallization texture.

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Discussion on this paper, TP 3549E, may be sent, 2 copies, to AIME by Dec. 1, 1953. Manuscript, Jan. 3, 1953. Cleveland Meeting, October 1953.

Table I. Composition of Crystal-Bar Zirconium

Element	Weight Pct
Hf	0.3 max.
Fe	0.02-0.1
Al	0.005-0.02
Cr	0.02-0.1
Cu	0.001-0.005
Ni	0.005-0.02
Mg	0.001-0.005
Mo	0.01
N_2	0.007*

* Chemical analysis (oxygen and carbon are estimated at 0.02 pct).

Experimental Procedure

The material used in this investigation was crystal-bar zirconium prepared by the iodide process. The major impurity was hafnium which was less than 0.3 pct. The composition obtained by spectrographic analysis is given in Table I.

The hot-rolled specimen was prepared from a 50-g button of zirconium arc-melted in a water-cooled crucible under an argon atmosphere. This button was enclosed in a welded sheath of soft iron and the ensemble was rolled at 825°C. After each pass the sheath was put back into the furnace to allow it to return to the desired temperature. The zirconium ingot was reduced in this manner to a thickness of about 0.035 in., a reduction of approximately 90 pct. The sheet after removal from the sheath was alternately polished and etched to a thickness of 0.001 in.

Cold-rolled specimens were prepared from crystal bar by a series of reductions of approximately 0.010 in. until the sheet reached a thickness of 0.010 in. after which it was cold-rolled to 0.001 in. thickness between sheets of alloy steel. The total cold reduction amounted to about 99.8 pct. Specimens were annealed in an evacuated Vycor or quartz tube.

The transmission pole figures were obtained by the quantitative method of Decker, Asp, and Harker[8] modified by Geisler[7] using an X-ray spectrogoniometer with Cu Kα radiation. Unless otherwise specified all four quadrants were examined with 5° increments along the radius of the pole figure. Absorption corrections were made for changes in length of the X-ray path through the sample as inclination of the

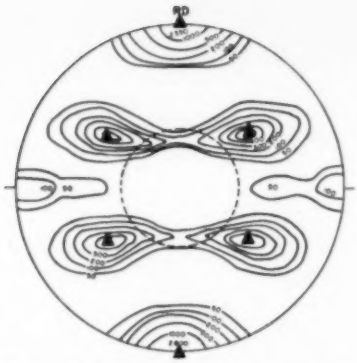


Fig. 1—$(10\bar{1}0)$ pole figure for 99.8 pct cold-rolled zirconium sheet. The orientations of $(10\bar{1}0)$ poles for the deformation texture (0001) $[10\bar{1}0]$ with a tilt of $\pm 40°$ about the axis of the rolling direction are indicated by triangles.

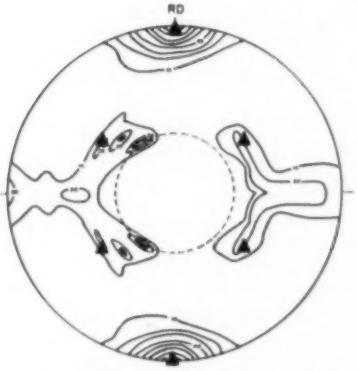


Fig. 2—$(10\bar{1}0)$ pole figure for zirconium sheet hot-rolled in a sheath at 825°C to a reduction of approximately 90 pct. Two quadrants examined. The orientations of $(10\bar{1}0)$ poles for the deformation texture (0001) $[10\bar{1}0]$ with a tilt of $\pm 40°$ about the axis of the rolling direction are indicated by triangles.

sample to the beam changed. During examination, the specimen was translated back and forth in its plane through a ½-in. distance in order to increase the total number of grains contributing to the measured X-ray intensity.

Fig. 3—Micrograph of hot-rolled zirconium. Electropolished. Polarized light. X250.

Results and Discussion

The pole figures for the cold-rolled, Fig. 1,* and for the hot-rolled, Fig. 2, zirconium showed the

* The contour units are arbitrary and are relative for a given pole figure. Units should not be compared from different pole figures.

$[10\bar{1}0]$ direction to be parallel to the rolling direction in agreement with McGeary and Lustman[5] and different from the findings of Burgers and Jacobs[4] that the "digonal axis I," the $[11\bar{2}0]$ direction, was in the rolling direction in cold-rolled zirconium. It was also observed that the texture is characterized by a tilt of basal poles toward the transverse direction with an average orientation of the planes at $\pm 40°$ from the rolling plane (indicating that the basal planes were tilted $\pm 40°$ from the rolling plane about the rolling direction). This 40° tilt is to be compared with the 30° tilt found by McGeary and Lustman.[5] Fig. 3 shows the microstructure of the hot-rolled zirconium.

Annealing at 300°C for 1 hr produced a texture

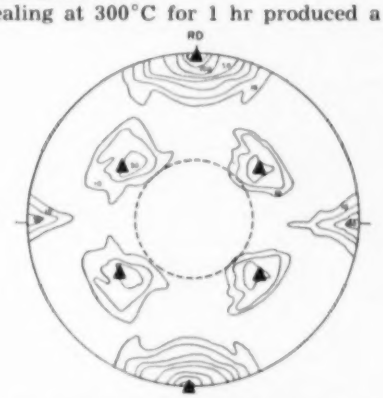


Fig. 4—$(10\bar{1}0)$ pole figure for 99.8 pct cold-reduced zirconium sheet annealed 1 hr at 400°C. Two quadrants examined. The orientations of $(10\bar{1}0)$ poles for the deformation texture (0001) $[10\bar{1}0]$ with a tilt of $\pm 40°$ about the axis of the rolling direction are indicated by triangles.

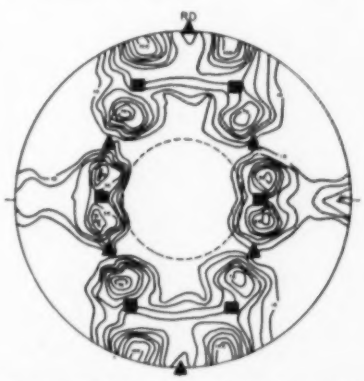


Fig. 5—$(10\bar{1}0)$ pole figure for 99.8 pct cold-reduced zirconium sheet annealed 1 hr at 500°C. Two quadrants examined. The orientations of $(10\bar{1}0)$ poles for the deformation texture (0001) $[10\bar{1}0]$ with a tilt of $\pm 40°$ about the axis of the rolling direction are indicated by triangles. The orientations of $(10\bar{1}0)$ poles for the recrystallization texture (000i) $[11\bar{2}0]$ with a similar tilt are indicated by squares.

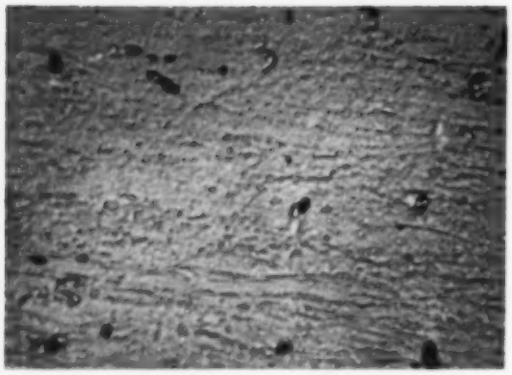


Fig. 6a—Micrograph of 97 pct cold-rolled zirconium sheet annealed 1 hr at 400°C. Longitudinal section. Bright field. Etched with HNO_3-H_2O-HF. X1000.

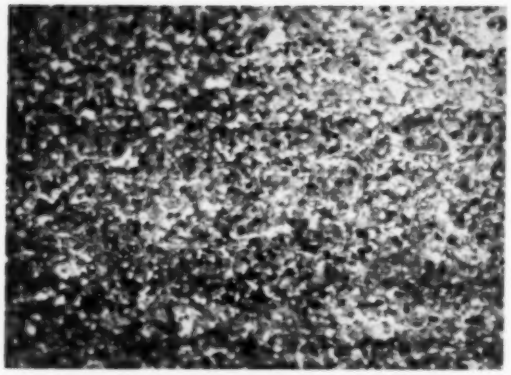


Fig. 6b—Micrograph of 97 pct cold-rolled zirconium sheet annealed at 500°C for 1 hr. Electropolished longitudinal section. Polarized light. X250.

which differed from the cold-rolled texture, Fig. 1, by having the four high-intensity regions near the center of the pole figure unconnected.

Annealing for 1 hr at 400°C produced a texture, Fig. 4, that was only slightly different† from that of the cold-rolled material. Metallographic examination disclosed the start of recrystallization as shown in the micrograph of Fig. 6a. It can be observed from the pole figure, Fig. 4, that the "fingers" extending from the high intensity areas are suggestive of the start of rotation which may be associated with the start of recrystallization. The change in texture from Fig. 1 to Fig. 4 is not necessarily a sharpening since, although the low intensity areas are smaller, the high intensity areas are not relatively more intense.

† The elongation of tensile samples annealed 1 hr at 400°C after a cold reduction of 97 pct was that of cold-rolled material although the yield strength has decreased slightly from that of the cold-rolled material.

The texture of rolled crystal bar annealed 1 hr at 500°C, Fig. 5, disclosed a double rotation about the poles of the basal plane placing the $[11\bar{2}0]$ direction in the rolling direction.

The texture after annealing 1 hr at 500°, Fig. 5, can also be described as a rotation of the cold-rolling texture, Fig. 1, of roughly 20° in either direction about the normal of the rolling plane. However, if this stage of the annealing is considered intermediate between the cold-rolling texture, Fig. 1, and the texture obtained at higher annealing temperatures, Fig. 7, rotation about the basal pole is more easily rationalized because the rotation about the normal of the rolling plane does not fit the textures obtained at higher annealing temperatures. Vestiges of the rolling texture remain as can be seen from the lower intensity $[10\bar{1}0]$ areas extending in both the rolling direction and in the transverse direction. This material‡ exhibited a very fine recrystallized grain size as shown in Fig. 6b.

‡ Tensile samples annealed 1 hr at 500°C after a cold reduction of 97 pct also indicated that recrystallization was not quite complete since the percentage of elongation was slightly lower and the yield strength slightly higher than the values obtained for material annealed at 600° or 700°C.

The texture of zirconium annealed 30 min at 600°C also indicated a double rotation of the texture had occurred during recrystallization, Fig. 7. From

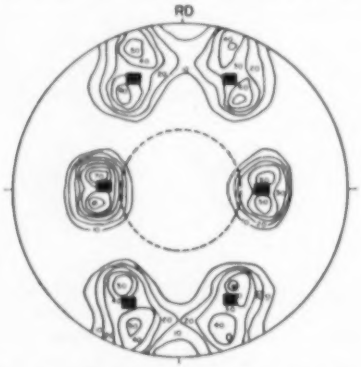


Fig. 7—$(10\bar{1}0)$ pole figure of 99.8 pct cold-reduced zirconium sheet annealed 30 min at 600°C. The orientations of $(10\bar{1}0)$ poles for the recrystallization texture (0001) $[11\bar{2}0]$ with a tilt of ±40° about the rolling direction are indicated by squares.

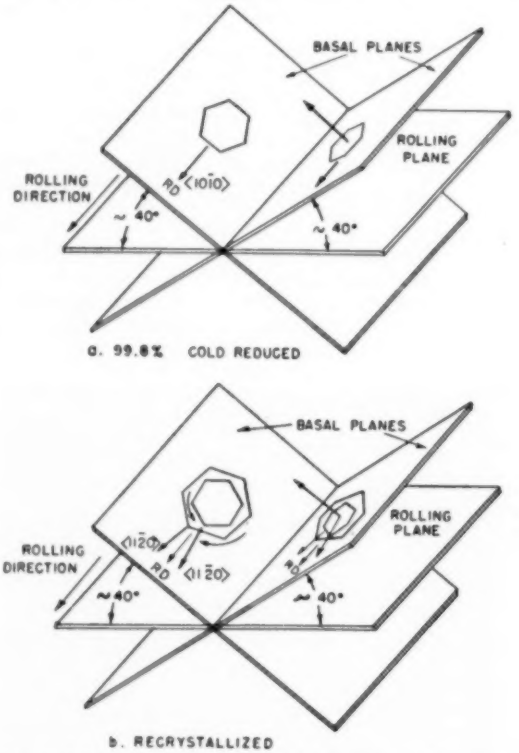


Fig. 8—Schematic representation of deformation and recrystallization textures found in zirconium sheet.

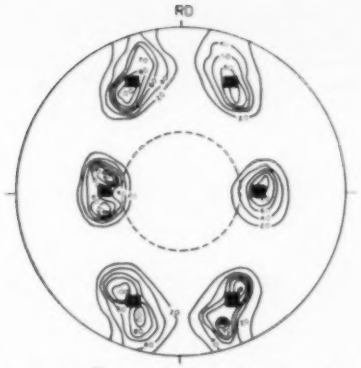


Fig. 9—(10$\bar{1}$0) pole figure of 99.8 pct cold-reduced zirconium sheet annealed 30 min at 900°C. The orientations of (10$\bar{1}$0) poles for the recrystallization texture (0001) [11$\bar{2}$0] with a tilt of ±40° about the rolling direction are indicated by squares.

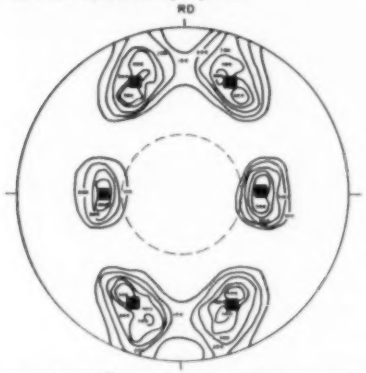


Fig. 10—(10$\bar{1}$0) pole figure of 99.8 pct cold-reduced zirconium sheet annealed 30 min at 900°C and furnace-cooled. The orientations of (10$\bar{1}$0) poles for recrystallization texture (0001) [11$\bar{2}$0] with a tilt of ±40° about the rolling direction are indicated by squares.

the data obtained for cold-rolled sheet annealed at 400°, 500°, and 600°C it is apparent that the deformation texture disappears during recrystallization and that a new texture with the [11$\bar{2}$0] direction in the rolling direction takes its place. This is different from the results reported for titanium[2] and beryllium[3] where the recrystallization texture was similar to the rolling texture.

The rotational difference between the deformation orientation and the annealing orientation is nominally 30° in agreement with McGeary and Lustman,[5] compare Figs. 1 and 7. However, measurement of the angle of rotation from the high intensity areas of the deformation texture to each of the double maxima in the annealing orientations showed that in detail the rotations appeared to be combinations of a small rotation of the range 16° to 24° and a larger rotation in the range 34° to 46°. The reasonable rotation combinations about the c-axis accounting for the change from the cold-rolled texture could be one or both of the following: 1—34° to 46° rotation in opposite directions, 2—16° to 24° rotation in opposite directions. From these rotations it follows that actually the [11$\bar{2}$0] directions of the recrystallized texture are still not in the rolling plane but tilted about 10° from the rolling direction (and out of the rolling plane) since the planes of rotation (tilt) are the basal planes that are about 40° from the rolling plane. Deformation and recrystallization textures are shown schematically in Fig. 8.

The transformation texture obtained, Fig. 9, by heating cold-rolled zirconium to 900°C, about 40°C above the allotropic transformation temperature, was found to be very similar to the recrystallization texture, in which the [11$\bar{2}$0] direction was in the rolling direction. The principal texture was sharper than that obtained at 600°C and the spread of the secondary double texture was reduced. This sharpening differs from the finding of McGeary and Lustman that the transformation orientation showed more scatter than that for annealing below the transformation temperature. It is not certain whether or not any recrystallization had occurred below the transformation temperature in the case of the transformation texture.

Fig. 9 shows the texture of 99.8 pct cold-reduced zirconium sheet annealed 30 min at 900°C and cooled fairly rapidly in the evacuated quartz tube, and Fig. 10 is the texture of similarly rolled and annealed sheet cooled slowly in the furnace. The texture shown in Fig. 11 is that of the specimen annealed at 600°C (texture shown in Fig. 7) and later annealed again for 30 min at 900°C. In all three instances the transformation textures are similar to the recrystallization texture showing a symmetrical texture having ±40° tilts of the basal planes out of

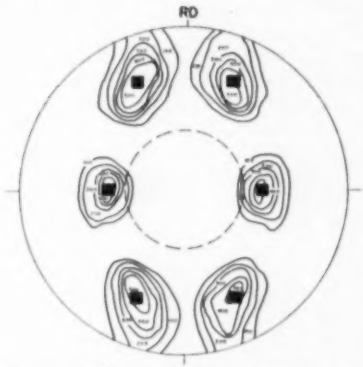


Fig. 11—(10$\bar{1}$0) pole figure of 99.8 pct cold-reduced zirconium sheet annealed first at 600°C for 30 min (Fig. 8) and later at 900°C for 30 min. The orientations of (10$\bar{1}$0) poles for the recrystallization texture (0001) [11$\bar{2}$0] with a tilt of ±40° about the rolling direction are indicated by squares.

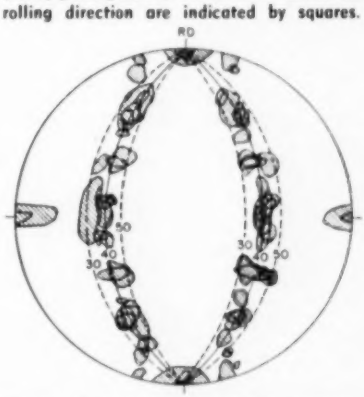


Fig. 12—Plot of locations of high intensity areas of (10$\bar{1}$0) poles showing concentration along 40° meridian.

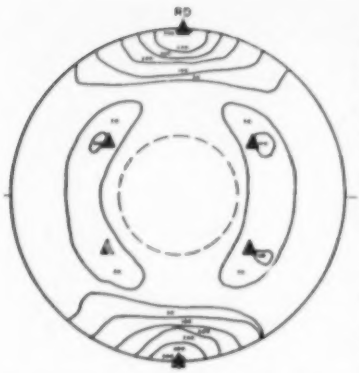
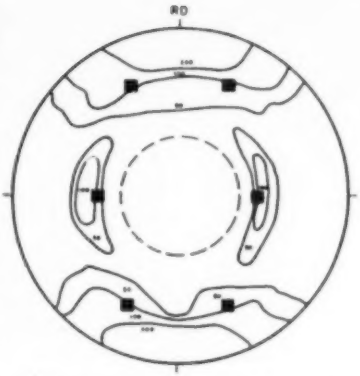


Fig. 13 (left)—$(10\bar{1}0)$ pole figure for 50 pct cold-reduced zirconium sheet prepared by cold reduction in 50 pct increments with intervening anneals for 30 min at 600°C. The orientations of $(10\bar{1}0)$ poles for the deformation texture (0001) $[10\bar{1}0]$ with a tilt of ±40° about the axis of the rolling direction are indicated by triangles.

Fig. 14 (right)—$(10\bar{1}0)$ pole figure for recrystallized zirconium sheet prepared by cold reduction in 50 pct increments with intervening anneals for 30 min at 600°C. As annealed. The orientations of $(10\bar{1}0)$ poles for the recrystallization texture (0001) $[11\bar{2}0]$ with a tilt of ± 40° about the rolling direction are indicated by squares.



the rolling plane and with the $[11\bar{2}0]$ direction approximately in the rolling direction.

It was observed that the texture of sheet zirconium that had been recrystallized and then annealed above the allotropic transformation temperature, Fig. 11, did not show the double maxima in the high-intensity areas exhibited by the other annealing textures.

The symmetrical texture at ±40° from the rolling plane continued to be present in the textures of annealed sheets, recrystallized and cycled through the allotropic transformation temperature. The location of high intensity areas of $(10\bar{1}0)$ poles along the 40° meridians for various cold-rolled and annealed textures is shown in Fig. 12.

Burgers[7] has pointed out that in the transformation of the β body-centered cubic zirconium to the α hexagonal close-packed form at approximately 860°C, the parallel planes for the transformation are the basal (0001) planes of the hexagonal close-packed form and the body diagonal (110) planes for the body-centered cubic modification. The parallel close-packed directions are the $[11\bar{2}0]$ direction of the hexagonal close-packed phase and the [111] direction of the body-centered cubic structure. These relationships would indicate that during transformation from the body-centered cubic modification there are 12 possible orientations which could be obtained in the hexagonal close-packed structure being formed. It is difficult to explain the return to the original texture after a recrystallized specimen has been heated and cooled through the transformation temperature unless some nucleating sites remain to cause the return of the body-centered cubic material during cooling to its original hexagonal close-packed orientation.

A strong texture in a material is often undesirable particularly when forming operations are planned. To obtain zirconium sheet with a weaker texture, several samples of crystal bar were rolled into thin sheet, 0.001 in. in thickness, by alternately cold reducing by 50 pct, and then annealing in vacuo for 30 min at 600°C. Pole figures from these specimens in the cold-rolled and in the annealed conditions are shown in Figs. 13 and 14. The sheet obtained in each case was more weakly oriented than the corresponding material prepared by cold-reduction of 99.8 pct and then annealing. Although the cold-rolled and the annealed specimens have somewhat similar textures, it can be seen that the $[10\bar{1}0]$ direction of the cold-rolled material and the $[11\bar{2}0]$ direction of the annealed material are approximately in the rolling direction. It can also be observed that the symmetrical texture produced by the 40° tilts of the basal plane out of the rolling plane is present in the material prepared by the cold-reduction and annealing cycle.

Summary

The deformation and annealing textures of iodide zirconium disclose a symmetrical orientation characterized by an average tilt of the basal planes ±40° out of the rolling plane toward the transverse direction and about the rolling direction.

The deformation orientations of cold-rolled and of hot-rolled material indicate the $[10\bar{1}0]$ direction to be parallel to the rolling direction.

The recrystallization texture is related to the deformation by a rotation about the (0001) poles which can be rationalized as a combination of 16° to 24° and/or 34° to 46° rotations, so that the $[11\bar{2}0]$ direction becomes approximately parallel to the rolling direction.

Working by alternately cold reducing and annealing produces a less highly oriented texture than does a single severe cold rolling and annealing.

Acknowledgment

The authors wish to thank Mrs. Ann S. Cooper who obtained the X-ray data and constructed the pole figures, and also other members of the Metallurgy Research Dept. who assisted in the experimental portions of this study. This work was supported by the U. S. Atomic Energy Commission under Contract No. W-31-109-Eng-52.

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Anelastic Behavior of Pure Gold Wire

by Donald R. Mash and Lewis D. Hall

The paper presents the results of experiments on the anelastic behavior of gold, as manifested by grain boundary relaxation. Two grain boundary internal friction peaks are found for 99.9998 pct Au. It is found that the peaks are associated with primary and secondary recrystallization. However, the existence of two discrete peaks cannot be explained on the basis of grain size and shape alone. It is suggested that grain boundary stability, as determined by orientation, plays a role in the observed effects.

EVIDENCE for the viscous behavior of grain boundaries in metals has been presented in recent years by several investigators, based upon studies of various anelastic effects, especially internal friction. Kê[1] has contributed greatly to this field, having put forward a coherent body of evidence for stress relaxation by the viscous intercrystalline flow mechanism. In this connection, he has made extensive use of pure aluminum (99.991 pct) as the test material, although he has also studied other metals and alloys, including pure iron (Puron).[2] Rotherham, Smith, and Greenough[3] have studied the internal friction of pure tin, interpreting their results in a manner similar to that of Kê. In view of the importance of such studies in shedding light upon the fundamental structure and behavior of the grain boundaries in pure metals, it appears that the use of a very pure test material which is inert to its environment should provide useful information on anelastic properties and the source of such behavior in pure metals.

The present work was carried out on spectrographically pure, 99.9998 pct Au, free of all impurities except for a trace of silver, estimated to be present to the extent of about 0.0002 pct. The term "pure gold" will hereafter refer to this very pure material. Gold of commercial purity, 99.98 pct, was also studied to observe the effects of small amounts of impurities. A pure gold "single crystal" specimen was also tested for comparison.

The variation of the internal friction and rigidity modulus as a function of temperature was determined by means of a torsion pendulum apparatus employing extremely low stress amplitudes and a frequency of vibration of the order of 1 cycle per sec. A 12 in. length of 0.031 in. (20 gage) gold wire formed the suspension element. The apparatus was similar to that described by Kê.[1] The test procedure and the basic requirements to be met for obtaining useful experimental data by this method have been given elsewhere.[1,2] It should be made clear that in all of the experiments to be described, the internal friction and rigidity were independent of the amplitude of torsional vibration.

The semilog plot of amplitude of vibration vs ordinal number of vibration was a straight line. This was carefully verified for each internal friction measurement. The linear variation shows that the internal friction was independent of stress; i.e., that the specimens were not being cold-worked during testing. The reproducibility of the internal friction curves, which were obtained by cyclic heating and cooling, indicates that the gold was unaffected by its environment during the tests.

The measure of internal friction adopted in the present study is the conventional "logarithmic decrement," defined as follows:

$$\text{log. dec.} = 1/n \ln A_o/A_n \qquad [1]$$

where n is the number of cycles or vibrations; A_o, the initial amplitude of vibration; and A_n, the amplitude after the nth cycle. When the logarithmic decrement is small, the shear modulus, G, of the wire is proportional to the square of the frequency of vibration provided the length and radius of the wire are kept constant. A plot of frequency squared vs temperature gives the following ratio:[1]

$$\frac{G_R}{G_U} = \left(\frac{f_p}{f_s}\right)^2 \qquad [2]$$

This expresses the fraction of the stress which has not been relaxed at a given temperature. G_R and G_U are the relaxed and unrelaxed moduli, respectively. The frequency of vibration in the polycrystalline specimen is f_p, and the frequency of vibration of a single crystal is f_s. This latter quantity is obtained simply by extrapolating the linear, low temperature portion of the curve of frequency squared vs temperature for the polycrystalline specimens.

The theory of viscous grain boundary stress relaxation as demonstrated by the anelastic behavior of metals has been discussed in detail by Zener[4] and need not be reproduced here.

Experimental Results

Initial measurements of the internal friction of pure gold were carried out on specimens which had been drawn with no intermediate annealing, resulting in a material which had undergone approximately 99 pct reduction of area in final processing. Annealing was then carried out at successively higher temperatures starting at 400°F for 1 hr and proceeding in this manner to as high as 1600°F in 100°F intervals. After each annealing treatment an internal friction and rigidity vs temperature curve was obtained over the range from room temperature to the particular annealing temperature. The resulting internal friction curves did not exhibit well defined maxima (peaks), but rather several fairly flat

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Discussion on this paper, TP 3536E, may be sent, 2 copies, to AIME by Dec. 1, 1953. Manuscript, Jan. 13, 1953. Cleveland Meeting, October 1953.

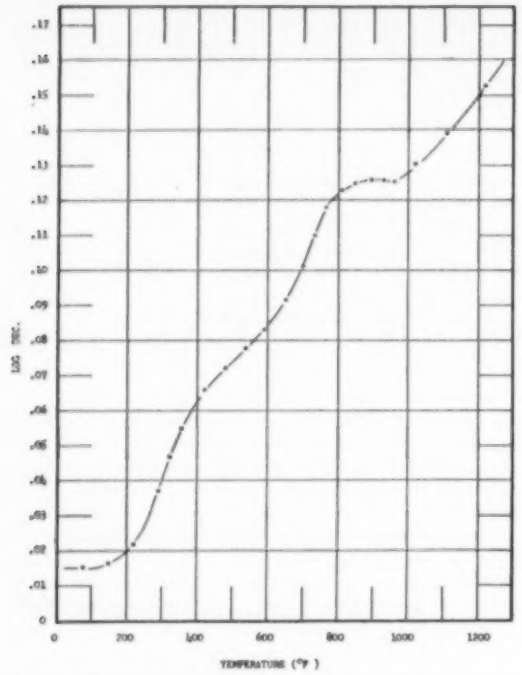


Fig. 1—Temperature variation of the internal friction of 99.9998 pct Au wire which had previously undergone 99 pct reduction of area. Specimen annealed 6.5 hr at 1600°F.

inflection points were obtained. For example, Fig. 1 shows that even after annealing for 6.5 hr at 1600°F no definite peaks were obtained in specimen No. 1, a result which is typical of heavily cold-worked material.

Tests on this material were, therefore, discontinued in favor of specimens which had undergone considerably less final cold working. Most of the work was performed on specimens which had been reduced in area about 36 pct in final drawing. Typical results for a series of annealing temperatures on the same test wire, No. 2, are shown in Figs. 2 and 3. In order to minimize confusion, the graphs contain only the results of anneals at 1000°, 1250°, and 1500°F. Curves were actually run for annealing temperatures at intervals of 100°F, from 400°F upward, as

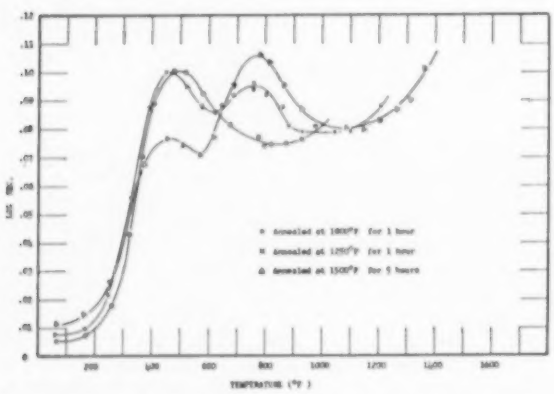


Fig. 2—The effect of annealing treatment upon the temperature variation of the internal friction of pure gold (specimen No. 2). Specimen had undergone 36 pct reduction of area prior to testing.

previously explained. A metallographic sample was taken after each anneal from specimens placed near the gold suspension wire in the furnace. This provided information concerning the microstructure associated with each annealing treatment and corresponding internal friction and rigidity curves. Fig. 4 shows some typical microstructures for specimen No. 2 after successive annealing treatments. These samples showed that after complete recrystallization had occurred at 600°F gradual grain growth continued until 800°F where an average grain diameter of about 0.105 mm obtained. This remained relatively constant up to 1000°F where "secondary" recrystallization was initiated; a few larger grains about 0.135 mm diam were observed. The curves shown in Figs. 2 and 3, after the 1000°F anneal are typical of the curves obtained at 800°F, also.

It can be observed that a single peak exists in the internal friction curve at about 500°F, while the point of inflection in the modulus curve[5] occurs at the same temperature. The peak is broad, indicating that it represents a wide distribution of relaxation times. The modulus curve shows a tendency to approach a value of G_R/G_U of about 0.80. This does not correspond closely to the theoretical value of 0.684 obtained by using the relation[1]

$$\frac{G_R}{G_U} = \frac{2(7+5\sigma)}{5(7-4\sigma)} \qquad [3]$$

and a value of 0.42 for Poisson's ratio, σ, of pure gold. Kê, in his extensive and widely quoted experiments with aluminum,[2] obtained almost perfect agreement of his experimental G_R/G_U ratio with that predicted by theory. It should be emphasized, however, that the equation for G_R/G_U is based on the assumption of elastic isotropy,[6] a condition which is closely approached in the case of aluminum but not in the case of gold. It is interesting to note that Rotherham, Smith, and Greenough,[6] working with tin, which is also quite anisotropic, found a similar discrepancy between theory and experiment. These results indicate that in comparing the very regular and orderly results of Kê with the behavior of other materials, it should be remembered that in few if any other systems is there a comparable degree of elastic isotropy. The deviations of any actual material from "ideal" behavior as regards anelastic properties must clearly be influenced by its degree of anisotropy. It should also be pointed out that the values of Poisson's ratio used in Eq. 3 are usually those for room temperature, whereas the relaxation occurs at elevated temperature. Since Poisson's ratio varies with temperature, calculated values of G_R/G_U will be correspondingly affected.

Annealing at 1250°F produced considerable grain growth, resulting in a microstructure containing an average grain size of about 0.195 mm diam. Secondary recrystallization had apparently been completed, Fig. 4. The internal friction curve is then found to consist of two peaks, separated by about 300°F (167°C), a rather surprising result and one which does not appear to have been observed before. Also, the background internal friction appears to have increased slightly, indicating that some other source of energy loss has become active. The lower temperature peak has become centered about 460°F and appears to be somewhat narrower than that due to the 1000°F anneal, indicating a more restricted range of relaxation times. The second peak, centered about 760°F, possesses a somewhat lower maximum value of the decrement and is rather broad. Anneal-

ing at 1500°F for 5 hr produced a relatively uniform grain size of about 0.270 mm. It can be observed from Fig. 2 that an inversion of the two peaks has occurred. The lower peak has decreased to a decrement of about 0.077, while the upper peak has reached a value of almost 0.11. Study of Fig. 3 shows that this sudden occurrence, and final predominance, of the second peak corresponds to the gradual broadening of the relaxation band of the dynamic modulus curve at 1250° and at 1500°F. The relaxation in the modulus from G_U to G_R now requires a much wider temperature range for its completion. However, after its initial decrease to about 0.80, the ratio G_R/G_U changes little, as shown by Fig. 3. This indicates that the same relaxation mechanism continues to prevail. In the modulus curve for the 1500° anneal a second, though slight, point of inflection has appeared at about 775°F, while the principal point of inflection occurs at about 475°F. The extreme breadth of the two internal friction peaks should also be noted.

It was desired to compare the activation energy values associated with the two maxima. This was done by means of the following relation:[3]

$$Q = \frac{R \ln (f_2/f_1)}{1/T_1 - 1/T_2} \qquad [4]$$

where Q is the activation energy, f_1 and f_2 are two different frequencies of vibration on the same test specimen, $(1/T_1 - 1/T_2)$ is the horizontal shift of one curve to superimpose it on the other when plotted against 1/°K as abscissa, and R is the gas constant. Fig. 5 shows the curve for specimen No. 3, also subjected to 36 pct reduction of area prior to testing, annealed at 1000°F for 1 hr in order to obtain one peak only. The value of Q was found to be about 34,500 cal per mol, which is accurate to about 10 pct. This specimen was annealed at 1500°F for 5 hr in order to produce the double peaks. From Fig. 6, the activation energy for the high temperature peak was found to be 58,000 cal per mol. It should be pointed out here that if the two peaks, high and low temperature, of one curve possessed similar activation energies, this superposition should have brought them both into coincidence with the double peaks of the other curve. This is clearly not the case. Specimen No. 4, possessing only about 17 pct reduction of area, was run in a manner similar to No. 3, already described. Similar results were obtained.

In order to evaluate the effect of the grain boundaries, a 10 in. long "single crystal" pure gold wire,

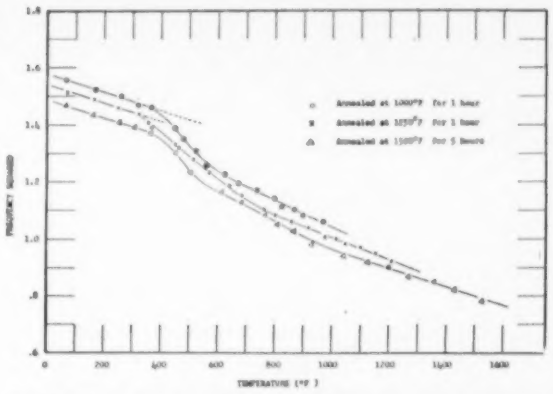
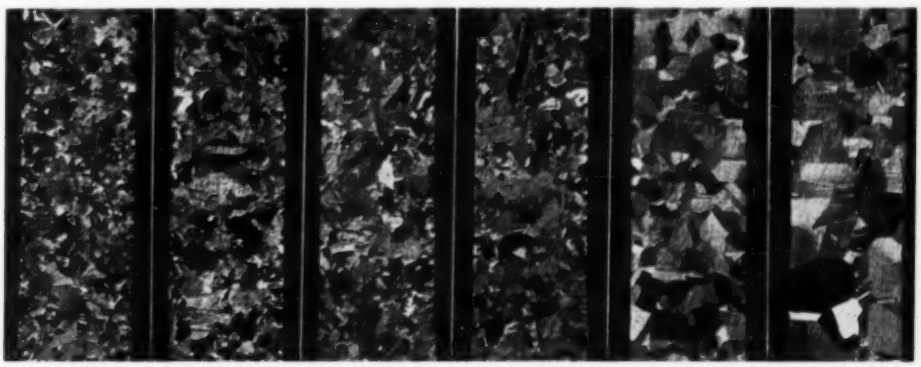


Fig. 3—The effect of annealing treatment upon the temperature variation of the "rigidity" of pure gold, specimen No. 2. Compare with Fig. 2.

approximately 0.040 in. diam, was produced. The entire specimen actually consisted of about three large crystals. The results of the test on this specimen are shown in Fig. 7. Unfortunately, in removing this specimen from the mold and subsequently placing it in the torsion pendulum apparatus, some cold working was unavoidable and is reflected in the curves of the internal friction and modulus vs temperature. The point of the sudden rise in internal friction in the specimen coincides rather closely with the position of the high temperature peak in the polycrystalline specimens. However, no peak is observed. Such behavior in single-crystal specimens which have undergone small amounts of cold work prior to testing has been observed by Kê[7] in the case of aluminum.

The great purity of the test material was thought to be a factor in revealing the double peak in the polycrystalline metal. Therefore, for purposes of comparison, a sample, No. 5, of commercially pure gold, 99.98 pct, possessing a final reduction of area of 36 pct was tested. Fig. 8 shows that no double peak was obtained. Instead, a single peak occurs at about 635°F which is intermediate between the low and high temperature peaks of the pure material. Annealing at 1500°F is seen to produce no result other than the movement of the peak to a higher temperature and a lower maximum value of the internal friction. The grain structure of the specimen after the two annealing treatments is shown in Fig.



600°F 700°F 800°F 1000°F 1250°F 1500°F

Fig. 4—Typical microstructures of pure gold specimens after treating at successively higher annealing temperatures. Compare with Figs. 2 and 3. X25.

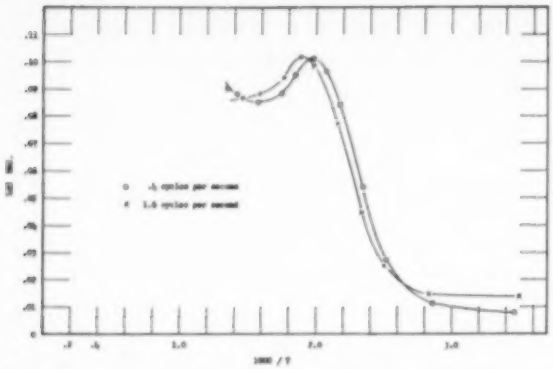


Fig. 5—Pure gold specimen No. 3 annealed for 1 hr at 1000°F to produce a single grain boundary peak. The activation energy is 34,500 cal per mol.

9. The 1000°F anneal produced a fairly uniform grain size of about 0.195 mm; the 1500°F treatment resulted in grains most of which were 0.360 mm in diam, although a few 0.270 grains were present. The value of G_u/G_r was found to be about 0.82, which closely corresponds to that of the purer material.

In an effort to throw some light on the source of this effect, the double peak, further experimental work was carried out. X-ray analyses by back-reflection and transmission techniques were carried out at different temperatures with pure polycrystalline specimens, treated comparably to No. 2. The transmission patterns, made with filtered CuKα radiation, on a specimen which was etched to 0.014 in. diam showed the existence of a recrystallization texture, approximately [211], in the interior of the wire, after annealing at 1000°F and below, which had almost disappeared after a 1600°F anneal. These patterns are shown in Figs. 10 and 11.

In considering possible sources of the anelastic effect which is manifested in the double peak, two possibilities may be eliminated at the outset. The stress-induced preferential orientation of foreign atoms cannot be a factor, since, as just pointed out, two peaks appear only in 99.9998 pct Au, while gold of 99.98 pct purity exhibits a single peak.

The possibility of a contribution to the internal friction by the motion of dislocations which have been introduced by cold work during the fabrication of the test wires is recognized. This, however, results in internal friction which increases continuously at high temperatures, with no tendency to form a peak. Further, the effect of prior cold work is to mask the grain-boundary peak or peaks, as shown by comparison of Figs. 1 and 2.

The behavior of both internal friction peaks in general, particularly the lower peak, corresponds to that which is predicted of the grain boundary as a relaxation center. They are found only in polycrystalline specimens, and not in the single-crystal specimen. With increasing grain size they move to higher temperatures, the lower peak being more strongly affected by this treatment than the upper. The curve of rigidity modulus vs temperature (Fig. 3) has its chief point of inflection at approximately the temperature of the lower peak, with a small, but perceptible second point of inflection at approximately the temperature of the upper peak. The broadness of the two peaks is reflected in the wide temperature range required for the complete relaxation of the modulus from its unrelaxed value, G_u, to its relaxed value, G_r.

The evidence cited indicates clearly that the lower peak is connected with grain boundary movement. For the upper peak, there is additional evidence linking it to this same phenomenon. First, the fact that the fraction of stress relaxation observed increased little after its initial drop to about 0.80, Fig. 3, argues in favor of the operation of a single relaxation process. If further relaxation arose from within the grains, the fraction would not remain roughly constant, but would increase markedly, due to the new contribution to the overall amount of stress relaxation. Furthermore, examination of the microstructure resulting from the 1500°F annealing treatment reveals a grain size which is still considerably smaller than the diameter of the test wire. It is, therefore, reasonable to expect that such a large volume of grain boundaries should contribute considerably more to the internal friction than that represented by the relatively small lower peak under these conditions.

Further consideration leads to the conclusion that grain size and shape are not the only factors leading

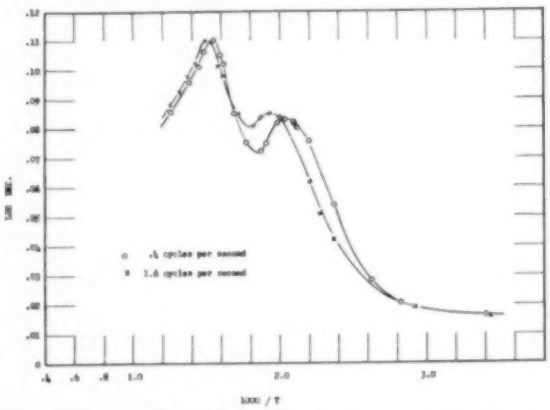


Fig. 6—Determination of the activation energy of the "upper" peak. Same specimen (No. 3) as Fig. 5, annealed for 5 hr at 1500°F to obtain the double peaks. A value of 58,000 cal per mol is obtained.

to a double grain boundary peak as opposed to a single peak. In fact, it is difficult to see a direct relation between cause and effect based only on grain size and shape. Careful scrutiny of the 1250° and 1500°F specimen in Fig. 4 does not disclose such an obvious degree of grain size contrast at either temperature as would account for a difference of some 300°F (167°C) between the low and high temperature peaks. This corresponds to a difference in relaxation time of about 0.01 sec, which is appreciable.

The explanation, on the basis of grain size, of the simultaneous existence of two peaks after annealing at intermediate temperatures, demands the existence of a marked duplex structure, with two widely different coexisting grain sizes. This is not observed. In the case of the 1500°F anneal, resulting in a uniform grain size, with almost no evidence of a duplex structure, only a single peak would be expected. However, the two peaks persist, although the lower peak has subsided considerably. The pronounced subsidence of the lower peak after annealing at 1500°F, and its further degeneration to a mere arrest in the curve after annealing at 1650°C, cannot be explained on the basis of grain size, since

the grain size of the specimen remains small in comparison with the specimen diameter.

The metallographic evidence shows that the lower peak is associated with primary recrystallization and the upper peak with secondary recrystallization. These latter phenomena are not yet well understood; therefore, only a qualitative explanation of the double peak effect in terms of them can be given. It is generally accepted that the driving force for secondary recrystallization is surface energy, i.e., the grains tend to adopt such configurations that the surface energy of their interfaces is a minimum. The change from primary to secondary structure thus entails an increase in boundary stability. The primary grains will thus have a certain grain boundary peak associated with them; a peak of lower activation energy because of the lower degree of boundary stability associated with this configuration. Upon secondary recrystallization, a structure with lower boundary free energy and greater boundary stability results. The grain boundary peak associated with this structure has a higher activation energy. Annealing at the highest temperatures results in almost complete disappearance of the primary structure and of the lower peak.

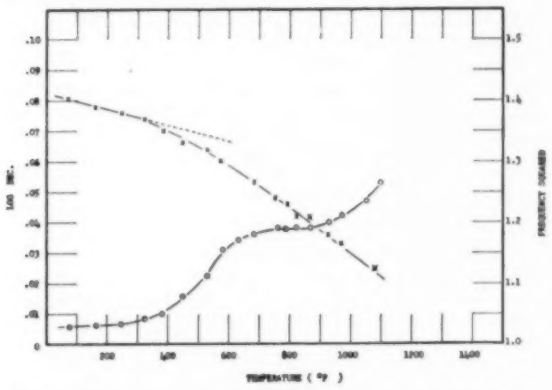


Fig. 7—Temperature variation of the internal friction and of the "rigidity" of a single-crystal specimen of pure gold.

These arguments are also confirmed by the X-ray evidence, which indicates the presence of a [211] recrystallization texture at 1000°F which has almost disappeared after annealing at 1600°F. Annealing at temperatures below 1000°F resulted in a single peak; annealing above 1000°F produced a second peak, the upper peak, and finally, annealing at about 1650°F (curve not shown) caused the lower peak to degenerate to an arrest in the curve, while the upper peak had decreased only slightly from its maximum height.

The upper peak was quite stable (resistant to further annealing treatments) but was also quite broad, indicating a wide range of relaxation times. This is rather remarkable in view of the fact that the grain size of such specimens was uniform, Fig. 4. On the basis of uniformity of grain size and shape alone, a narrow peak, such as was found in the commercially pure gold, would be expected. This lends additional support to the contention that other factors are of importance in grain boundary internal friction.

The question arises as to why no other investigators have reported a double grain boundary peak. This may be a result of: 1—degree of purity of materials used, and/or 2—maximum annealing

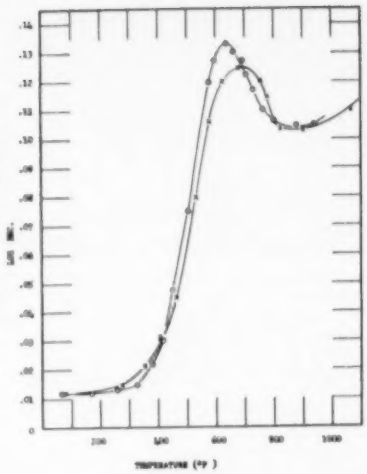


Fig. 8—Internal friction vs temperature for a commercially pure, 99.98 pct Au specimen, No. 5. Specimen had previously undergone 36 pct reduction of area. Circle annealed at 1000°F for 1 hr; cross annealed at 1500°F for 5 hr.

temperature employed in the course of measurements. As stated earlier, those impurities present in gold of 99.98 pct purity were sufficient to eliminate the double peak, which was intermediate in position between the two peaks of the 99.9998 pct Au. These results show the powerful effect which trace amounts of impurities can have in masking certain physical phenomena. They accent the desirability of working with materials of the highest possible purity and maximum resistance to contamination by their environment.

Kê[8] reports work on 99.999 pct Cu in which only one peak was observed, which would seem to cast some doubt on the first reason mentioned above. However, his maximum annealing temperature was 600°C (1112°F) and it is not known whether annealing above that temperature would have produced a second peak. It should be remembered that the upper peak in 99.9998 pct Au did not appear until the specimen had been annealed at 1250°F. In the case of other investigators and other materials, the purity of the specimens used has been comparable to the 99.98 pct Au, which showed only a single, narrow peak.

The maximum annealing temperature has an influence in determining the orientations existing in the material and the presence or absence of secondary recrystallization. Usually, if not always, in the case of other investigators this temperature has not been high enough to alter the deformation texture appreciably. For example, Barrett[9] states that the deformation texture of 99.996 pct Cu, which is [111], [100], is not altered by annealing at and below 500°C and retains the [111] component at 1000°C. He also states that the deformation texture of aluminum is not altered by annealing below 500°C. Kê, in his extensive experiments with aluminum, used a maximum annealing temperature of 450°C.

The failure of other investigators to detect a double peak lends support to the contention that orientation is a factor, since the grain sizes employed by them (e.g., see Kê[1]) have been comparable to those used in the present work. It is interesting to note that no detailed studies have been reported to date on the effect of grain orientation on such processes as grain boundary relaxation, grain boundary diffusion, and similar grain boundary phenomena. In this connection, it would be interesting to investigate the difference in grain

Fig. 9—Microstructures associated with curves shown in Fig. 8 for commercially pure gold specimen. Compare also with Fig. 4. X25. Area reduced approximately 50 pct for reproduction. a (upper)—1000°F. b (lower)—1500°F.

boundary relaxation between specimens prepared in various ways so as to produce various types of preferred orientation.

Summary

The anelastic behavior of grain boundaries in pure (99.9998 pct) gold was investigated by studying the variation of the internal friction and shear modulus in torsional vibration, using frequencies of the order of 1 cps. For comparison, commercially pure (99.98 pct) gold was also investigated, as well as a pure gold single-crystal specimen.

After annealing at temperatures between about 1200° and 1600°F, the curves of internal friction vs temperature of measurement for polycrystalline pure gold specimens exhibit two discrete grain boundary peaks at 460° and 760°F, with activation energies of 34,500 and 58,000 cal per mol, respectively. This effect has not previously been reported in the literature for any material. Only one peak appears in pure gold after annealing at temperatures outside this range. Commercially pure (99.98 pct) gold showed only one peak regardless of annealing temperature.

It is suggested that grain boundary stability, as determined by orientation, plays a role in the observed effects, and that grain size and shape are not the only factors involved. This conclusion is based on the following observations: 1—the association of the peaks with primary and secondary recrystallization; 2—the absence of duplex grain structure; 3—the considerable difference in the temperatures and activation energies of the two peaks; 4—the greater breadth of the peaks compared with the single peak in 99.98 pct Au of the same grain uniformity; 5—the marked subsidence of the lower peak after annealing at high temperatures, despite a grain size which is still small compared with the specimen diameter; 6—the correlation of the peaks with the presence and breakdown of a [211] wire texture.

Trace amounts of impurities have a remarkably strong influence in eliminating the double peak and reducing the width of the single peak that results. The impurities present in 99.98 pct Au as compared with 99.9998 pct Au (in which the double peak was observed) were sufficient to eliminate the double peak and to reduce greatly the width of the resulting single peak. This effect, in addition to the fact that high annealing temperatures are necessary to cause a second peak to appear in the pure material, probably accounts for the fact that other investigators have not observed the phenomenon.

The experimental results point up the advisability of considering the effect of grain orientation in processes which take place at grain boundaries, as well as the necessity of taking elastic anistropy into account when comparing the anelastic behavior of different materials.

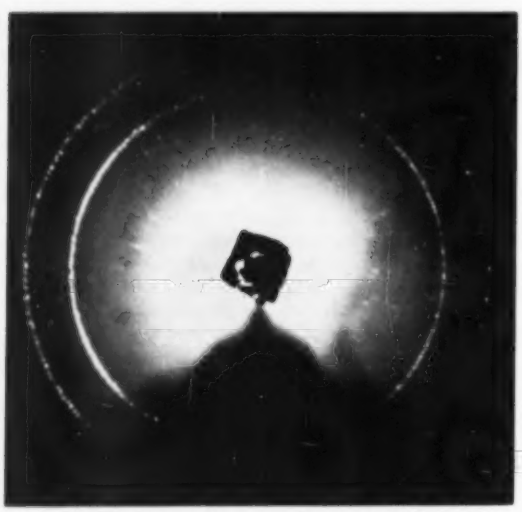


Fig. 10—Orientations after annealing at 1000°F. Texture: scatter about [211].

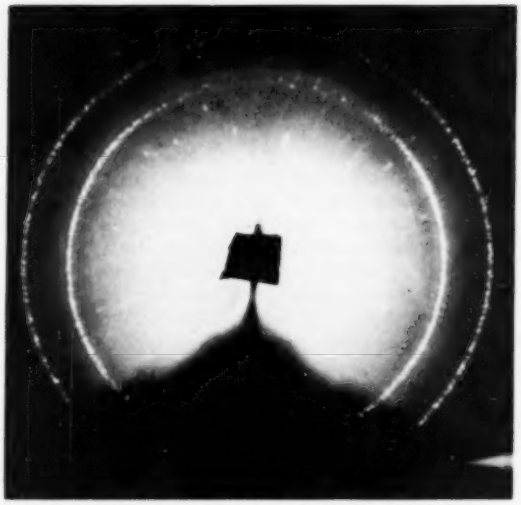


Fig. 11—Orientations after annealing at 1600°F. [211] texture has practically disappeared.

Acknowledgments

This investigation was sponsored by the Office of Naval Research, Contract N6-ori-154 Task V, Project N6-031-012.

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aime NEWS

New England Regional Conference Featured GE Laboratory Tour

One of the outstanding features of the recent New England Regional Conference was the conducted tour through General Electric Co.'s Schenectady, N. Y. Research Laboratory.

The laboratory overlooks the Mohawk River, with the Helderberg Mountains looming to the west. Vermont's Green Mountains, the Adirondack foothills, and the Catskill Mountains complete the mountainous ring surrounding the laboratory.

The tour included a resume of the work currently going on at the laboratory. Refreshments were served.

Fifteen groups of ten persons each accompanied by two escorts visited the following sections; chemistry stockroom, transformation laboratory, magnetics laboratory, structures laboratory, metallography laboratory, mechanical testing laboratory, heat treating laboratory, metal forming laboratory, electrical products shop, and the glass shop.

Among the demonstrations was seeding simulated clouds supercooled one which showed snow making by in a storage box.

Registrants, above, take a break between sessions at the General Electric Research Laboratory, Schenectady.

The New England Regional Conference Committee, left, responsible for the success of the meeting was, seated, A. I. Blank, W. H. Sharp, J. H. Moore, W. L. Stearns. Standing, T. W. Landig, M. B. Bever, L. P. Tarasov, F. Wilson, L. W. Thelin, and W. D. Robertson.

Division Officers Nominated for 1954

Institute of Metals Div.

Chairman: J. H. Scaff, Bell Telephone Laboratories; Senior Vice-Chairman: J. S. Smart, Jr., American Smelting & Refining Co.; Vice-Chairman: C. S. Barrett, Institute for the Study of Metals, University of Chicago; Secretary: Ernest Kirkendall, AIME. Executive Committee (three years): W. J. Harris, Jr., Minerals & Metals Advisory Board, National Research Council, National Academy of Sciences; J. E. Burke, General Electric Co.; J. M. Hodge, U. S. Steel Corp.

Iron and Steel Div.

Chairman: J. S. Marsh, Bethlehem Steel Co.; Vice-Chairman: G. B. McMeans, Kaiser Steel Corp; Vice-Chairman: Shadburn Marshall, U. S. Steel Corp.; Vice-Chairman: A. W. Thornton, U. S. Steel Corp. Executive Committee (three years): S. J. Dougherty, Weirton Steel Co.; John Chipman, Massachusetts Institute of Technology; K. L. Fetters, Youngstown Sheet & Tube Corp.

Extractive Metallurgy Div.

Chairman-Elect: A. W. Schlechten, Missouri School of Mines; Secretary: Ernest Kirkendall, AIME. Executive Committee (three years): H. H. Kellogg, Columbia University; J. G. Leckie, American Smelting & Refining Co.

(The current Chairman-Elect, P. T. Stroup, Aluminum Co. of America, automatically becomes Chairman of the Division.)

Mineral Industry Education Div.

Chairman: J. R. Van Pelt, Montana School of Mines; Vice-Chairman: J. W. Stewart, University of Alabama. Executive Committee (three years): R. E. Kirk, U. S. Steel Corp.; E. P. Pfleider, University of Minnesota; W. T. Thom, Jr., Princeton University; Past Chairman (non-elected): H. H. Power, University of Texas; Secretary: J. D. Forrester, Missouri School of Mines.

H. DeWitt Smith Heads 1954 AIME Nominations Slate

For President

H. DeWitt Smith, mining engineer, Newmont Mining Corp., New York, has been nominated for the Presi-

H. DeWITT SMITH

dency of AIME in 1955. Mr. Smith was born at Plantsville, Conn., and graduated from Hartford Public High School. In 1908 he received his Ph.B. from Yale University and his E.M. in 1910. After graduation from college he joined J. E. Spurr, Inc., as an engineer, and worked in the western United States and Mexico. From 1914 to 1917 Mr. Smith was foreman, mine superintendent, and assistant manager, successively, for Kennecott (Alaska) Copper Corp. He then joined the United Verde Copper Co., Jerome, Ariz., as mine superintendent. He later became general superintendent and was sales manager, New York City, from 1927 to 1930. Thereafter Mr. Smith was an engineer in the industrial dept. of the New York Trust Co. for three years. He then became associated with the Newmont Mining Corp. and has been a director since 1930 and a vice-president since 1946. He is also a director and member of the executive committee, Phelps Dodge Corp. and St. Joseph Lead Co. Mr. Smith is managing director of the O'okiep Copper Co., Ltd., and Tsumeb Corp., Ltd. He was adviser, vice-president and executive vice-president, Metals Reserve Co., a subsidiary of RFC, from 1941 to 1944. At this time Mr. Smith had been a Director of AIME, but resigned when he joined Metals Reserve Co. He was also Chairman of the AIME Nominating Committee in 1939. For nine years he has been a member of the Seeley W. Mudd Memorial Fund Committee and is now Chairman. In 1949 Mr. Smith was a Vice-Chairman of the 75th Anniversary Committee, AIME. He is a past president of the Mining & Metallurgical Society of America and also of the Mining Club.

For Vice-Presidents

Theodore Benton Counselman has been nominated as a Vice-President and Director of AIME. Mr. Counselman is manager of FluoSolids sales for The Dorr Co., Stamford, Conn. Columbia University gave him his E.M. degree in 1907 and he then began his career in the test mill of the Inspiration Copper Co. For a few years he was associated with the Cananea Consolidated Copper Co. and from 1912 to 1915 was a metallurgist for the Arizona Copper Co., Ltd. Mr. Counselman was engaged in research work for the Miami Copper Co. for one year and later worked in Minnesota for the Mesabi Iron Co. He became sales engineer with the Celite Products Co., Chicago in 1923. For two years he was Western representative for the Clark Car Co. and then joined Fort Pitt Mine Equipment, Pittsburgh, as sales manager. Mr. Counselman joined The Dorr Co., and has been sales engineer; manager, industrial div.; manager, contract engineering div.; and manager, FluoSolids sales, respectively. Among his inventions are magnetic separators, equipment for manufacturing synthetic rubber, and classifiers. He has written numerous technical articles on handling of flue dust in blast furnace plants, concentration of low grade iron ores, and manufacturing of cement. Mr. Counselman has been active in AIME as Chairman of the Chicago and New York Sections, Blast Furnace and Raw Materials Vice-Chairman, and Iron and Steel Div. Vice-Chairman. He has also served on awards and admission committees. For the last three years he has been Chairman of the National Membership Committee. Mr. Counselman is also a Member of the Executive Committee of the Institute and was named a Director in 1952. He was recently appointed a Vice-President for a one-year term. He holds membership in various professional societies and clubs.

T. B. COUNSELMAN

H. DECKER

Harold Decker, president, Houston Oil Co. of Texas, has been nominated for Vice-President and Director of AIME. Mr. Decker received his college education at Kenyon College, Gambier, Ohio, and the University of Oklahoma. He has also attended the Advanced Management Program of the Harvard University Graduate School of Business Administration. He has been associated with the Skelly Oil Co. and also the Seaboard Oil Co. In 1945 he joined the Pan American Production Co. as assistant manager and was promoted to vice-president and general manager. He resigned from this firm in 1950 to accept the presidency of Houston Oil Co. He is also president of the East Texas Pulp & Paper Co. Mr. Decker is a director of Halliburton Oil Well Cementing Co., Duncan, Okla., and of the South Texas National Bank, Houston. He is also a trustee of Southwest Research Institute and the Institute of Inventive Research, San Antonio. For years he has been active in various oil industry organizations. He

S. S. Clarke, Chairman of the Nominating Committee, has announced nominations for President-Elect, Vice-Presidents, and Directors for 1954. As provided in Art. IX, Sec. 2, of the bylaws, 25 Members or Associate Members may sign and transmit to the Secretary's office prior to Sept. 1 "any complete or partial ticket of nominees," should they wish other candidates to be considered.

In such instance, a letter ballot will be forwarded to all Members in good standing in the United States, Canada, and Mexico, tabulating both the official ballot and any supplementary nominations. If no supplementary nominations are thus received, no letter ballot will be printed, and nominees on the official ballot shall be declared duly elected at the meeting of the Board of Directors or the Executive Committee in November.

holds membership in the API and is a director of the Texas Mid-Continent Oil and Gas Assn. Mr. Decker is a director of the Independent Petroleum Assn. of America and vice-president of the Southeast Texas District. In 1947 he was Chairman of the AIME Gulf Coast Section. In 1948 and 1949 he was a Member of the Petroleum Branch Executive Committee and has been a Director of AIME since 1950.

For Directors

E. C. Babson, who has been nominated a Director of AIME for a three-year term, has been manager of the Canadian div. of Union Oil Co. of California, Calgary, Alberta, since 1951. Born in Los Angeles, he is a graduate of Stanford University, with an A.B. in 1930 and an M.E. in 1933. On graduation in that year he joined the Union Oil Co. of California as a research chemist at Wilmington, Calif. Mr. Babson has held various positions with this company. From 1936 to 1944 he was production engineer at Compton, Los Angeles, and Santa Fe Springs, Calif. For two years he was a lecturer in metallurgy in a war training program at the University of Southern California. Mr. Babson was made district production foreman for Union Oil in 1944. He became staff engineer at Whittier, Calif. in 1948, and in 1949 was appointed assistant chief petroleum engineer, Los Angeles. In 1951 he was acting chief, reserves section, production div. Petroleum Administration for Defense.

E. C. BABSON

G. D. DUB

George D. Dub, assistant to the president, Cyprus Mines Corp., Los Angeles, has been nominated Director of AIME. Mr. Dub received his B.S. from City College, New York, and his E.M. from Columbia School of Mines. From 1912 to 1925 Mr. Dub was employed as a mining engigineer by various companies. He has been located in Arizona, Montana, New Jersey, Washington, D. C., Chile, and Peru. In 1926 he was superintendent, general manager for the Pacific Alkali Co. and Pittsburgh Plate Glass Co., Bartlett, Calif. For approximately five years, Mr. Dub was a consulting engineer in Los Angeles and in 1951 joined Cyprus.

Ralph E. Kirk, manager of raw materials, Tennessee Coal & Iron Div., U. S. Steel Corp., Fairfield, Ala., has been nominated as a Director of AIME for a three-year term. Mr. Kirk joined U. S. Steel in 1936 and was appointed to his present position in 1943. Born in Luthersburg, Pa., he is a graduate of Pennsylvania State College. Mr. Kirk was superintendent of the Luzerne Coal & Coke Co. from 1914 to 1916 and then joined the H. C. Frick Coke Co. as superintendent. Mr. Kirk joined the Army during World War I and, after his discharge with the rank of captain, he rejoined the Frick Coke Co. In 1930 he accepted the position of general superintendent, Mahanoy div., Philadelphia & Reading Coal & Iron Co. He holds membership in several professional societies and had been a Chairman of the AIME Coal Div.

R. E. KIRK

C. C. LONG

Carleton C. Long has been nominated for Director of AIME for a three-year term. Dr. Long was Chairman of the Metals Branch Council in 1951, Chairman of the Extractive Metallurgy Div. in 1950, and served as Chairman of the Lead-Zinc Committee in 1948. Born in Boulder, Colo., Dr. Long received his B.S. in chemical engineering from the University of Colorado in 1931. In 1932 he received an M.A. in chemistry from Stanford University and a Ph.D. from Colorado in 1935. He has been associated with the St. Joseph Lead Co. zinc smelting div. since 1935. He started as a research engineer at the Josephtown smelter. In 1937 he was appointed director of the plant research dept., Monaca, Pa., a position he still holds. Dr. Long has made contributions to the literature of the electrothermic winning of zinc and is a member of several professional societies.

Earl Robert Marble, currently manager of the Tacoma smelter, American Smelting & Refining Co., Tacoma, Wash., has been nominated as a Director for three years. A native of Attleboro, Mass., Mr. Marble graduated from Tufts College in 1908. Prior to joining AS&R in 1914, Mr. Marble was a chemist with the Nevada Douglas Copper Co., Ludwig, Nev. His first position with AS&R was that of chemist at Hayden, Ariz. He was promoted to superintendent in 1923 and transferred to El Paso in 1927 as general superintendent. He held the same position at Tacoma until 1947 when he was appointed manager. Mr. Marble is a director of various organizations including the Northwest Mining Assn., Assn. of Washington Industries, and Tacoma Chamber of Commerce.

E. R. MARBLE

P. J. SHENON

Philip J. Shenon has been nominated as an AIME Director for a three-year term. Dr. Shenon was recently named a director of Day Mines, Inc. He is an authority on the Coeur d'Alene silver belt. Dr. Shenon has been in the employ of Day Mines since shortly after the end of World War II as a consulting geologist. He was born at Salmon, Idaho and is a graduate of the Universities of California, Idaho, and Minnesota. Following graduation he was employed as a geologist for the San Francisco Mines of Mexico, Continental Oil Co., and Idaho Bureau of Mines, successively. In 1928 he joined the staff of the Montana School of Mines as an assistant professor of geology. Two years later Dr. Shenon became associated with the U. S. Geological Survey and worked in western mining districts. He then worked in Canada as exploration geologist for the International Nickel Co. of Canada and returned to the United States in 1942 as consulting geologist. During World War II he served in the topographical mapping section of the Army. After his discharge he was named dean of the School of Mines, University of Utah. He resigned in 1952 to set up private consulting practice. Dr. Shenon was Chairman of the AIME Columbia Section in 1947, Chairman of the Mining Geology Committee in 1948, and was first Chairman of the Mining, Geology and Geophysics Div. in 1949 and 1950. Dr. Shenon was active in the formation of the latter division. He was a Member of the Mining Branch Council in 1950 and 1951. He holds membership in professional societies in the United States and Canada.

Pacific Northwest Metals and Minerals Conference

Attendance Typifies Region's Industrial Growth

More than 200 AIME members, ladies, and students turned out to make the second Annual Pacific Northwest Metals & Minerals Conference at Seattle's Benjamin Franklin Hotel May 14 to 16 one of the outstanding events of the year.

Field trips, technical sessions, and the Grand Banquet Saturday evening contributed to a full three-day meeting. Field trips held during the first day of the conference offered delegates a choice of Northwest Glass Co., and Bethlehem Pacific Coast Steel Corp.; and Tacoma smelter, American Smelting & Refining Co.

Women's Auxiliary activities started with a coffee hour following registration. Luncheons, shopping tours and special events filled mornings and afternoons. At the unusual Penthouse Theatre on the University of Washington Campus, the ladies saw Shakespeare's *Loves Labours Lost*.

Earl Marble, Director-Elect of the AIME, acted as toastmaster at the Grand Banquet, introducing George McMahon of the Pacific Petroleum Co., who spoke on oil and gas in the Pacific Northwest.

George G. Vincent, Aluminum Co. of Canada, told of his firm's progress in its British Columbia project, at the Metals Luncheon. Currently, the major undertaking at Kitimat is the construction of pot line buildings. Another activity during 1953 will be construction of a two-span bridge more than 600 ft long over the Kitimat River. Toastmaster for the luncheon was James G. Johnston.

Six papers were presented at the Iron and Steel Div. session. Four of the papers covered new practices for steel production and two dealt with advanced ideas on steel heat treating practices.

A. L. Ascik, metallurgist for Pittsburgh Metallurgical Co., discussed *Deoxidation Vs Degassification of Alloy Steels Made in Basic Electric Furnaces*. Mr. Ascik stressed the importance of superheating to increase oxygen solubility and in turn decreasing the hydrogen content of the melt to a harmless level. Desirability of subsequently cooling the metal before adding the final deoxidizers was discussed.

Vincent E. Beluske presented a paper on sampling methods at the furnace for composition control. The Brinell hardness test for making preliminary carbon determinations at the furnace on the hardenable grades of steel was investigated. Mr. Beluske described design and operation of a gun for sucking a metal sample for chemical analysis into a glass rod; a slag viscometer test; and a fluidity test for temperature control. Use of these control methods at the furnace were shown to shorten time required for a heat of steel, increasing tonnage per day.

K. W. McGrath, metallurgist, Atlas Steel Foundry & Machine Co., Tacoma, Wash., traced development of the fluidity test as a guide to metal temperature control for casting production. G. F. Heffernan, general superintendent, Vancouver Steel, Ltd., Vancouver, B. C., recounted experience with highly refractory roof brick of the mullite type. He demonstrated not only the economy of this refractory brick because of improved roof life, but that attendant improvement in life of the basic sidewalls represented a substantial saving and increase in period of operation between shut-down for relining. (See p. 884).

Some Short Cuts in the Heat Treatment of Steel Castings was offered by Alexander Finlayson, Seattle consulting metallurgist. Speed of transformations was discussed. In addition it was pointed out that many specifications for heat treating were superfluous in regard to required holding times at hardening temperatures. It was also noted that in tempering a short time at elevated temperatures could produce, in most cases, at least equal properties to those resulting from longer periods at lower temperature. Value of quenching from the tempering temperature to avoid temper brittleness was touched upon by Mr. Finlayson.

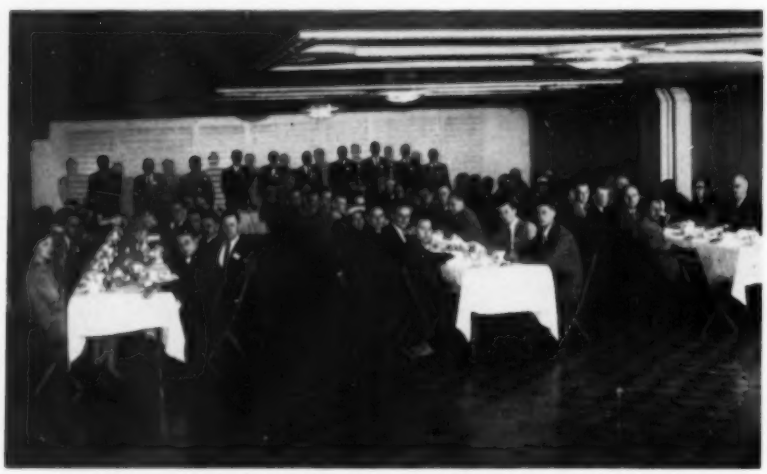
F. A. Schaufelberger, Chemical Construction Co., New York, and F. R. Morral, Kaiser Aluminum & Chemical Corp., Spokane, reported on decarburization of steel in molten chloride salt baths. The paper dealt with problem of moisture and humidity causing decarburization of the steel being heat treated and indicated means for keeping salt bath neutral to the immersed steel.

Paul T. Benson, general superintendent of the Tacoma smelter, presided over the Extractive Metallurgy meeting. Fve papers were presented. E. D. Dilling, Bureau of Mines at Albany, Ore., described zirconium production. He employed numerous slides in elaborating on his subject, showing several electric furnaces and zirconium tools. A serious corrosion problem was solved by the Bureau with fabrication of a valve for the hydraulic system of a fork truck.

Trials and tribulations of placing in operation the new Kaiser Aluminum plant at New Orleans were recounted by C. P. Love, manager of the Kaiser Tacoma plant. He also told of the South's great industrial expansion.

G. E. Sigler spoke on the history and development of the Tacoma smelter, American Smelting & Refining Co., since its beginning as a lead smelter in 1890. He told of the great period of expansion following the plant's acquisition by AS&R in 1906.

O. T. Gallagher presented a thought provoking paper on the safety dept. at the Tacoma smelter. Safety is everyone's job, from management on down to the lowest levels, he said.



One of the most successful social events of the Pacific Northwest Conference was the Metals Branch Luncheon. Some 99 persons attended and heard George G. Vincent's account of the aluminum project at Kitimat, British Columbia. James G. Johnston was luncheon toastmaster.

E. G. N. Player, assistant superintendent, Tadanac dept., Consolidated Mining & Smelting Co., Trail, B. C., discussed the problem of finding a use for sulphur dioxide. His firm is now selling as fertilizer the same material farmers once said was burning up their crops.

First paper presented at the Physical Metallurgy session, *High Temperature, High Pressure Oxidation of Metals II Tantalum in Oxygen*, by W. M. Fassell, Jr., and William McKewan, University of Utah, reported that tantalum oxidizes according to linear law and three oxidation rate processes may be involved.

O. G. Paasche, Oregon State College, described the equilibrium system of binary alloys of zirconium and silver in the composition range of 0 to 45 pct silver in his paper *The Zirconium-Silver System*. Co-author was R. S. Kemper.

A theory explaining observed phenomena in which self-lubricated alloys have resistance to accelerated-mechanical-abrasion which has been impossible to duplicate by external lubrication, was proposed by Harry Czyzewski, Metallurgical Engineers, Inc., Portland, Ore. Title of his paper was *Mechanical Aspect of Seizing in Metal Wear*. A series of manganese rich ferromagnetic alloys developed at the University of British Columbia by R. Butters and William Armstrong were discussed by Mr. Butters in the paper *Some Manganese Base Ferro Magnetic Alloys*. These alloys are based on an interstitial solution of carbon in face centered cubic manganese or alloys of manganese and certain non-transitional metals as aluminum, indium, tin, and zinc.

James I. Mueller described the new tool available to chemists, metallurgists and ceramists, X-ray spectrography. Qualitative and quantitative determinations are readily made using the X-ray technique.

Metallurgical aspects involved in design, fabrication and operation of gas turbines were described by W. L. Slosson of Boeing Airplane Co. He paid particular attention to those dealing with material properties, creep-rupture strength, short time tensile strength, fatigue strength, thermal expansion, thermal conductivity, thermal shock resistance, oxidation resistance and corrosion resistance, which must be gained using minimum amounts of strategic materials.

D. Vasarhelyi of the University of Washington spoke on *Plastic Strain and Fracture in Structural Steel Plates with Openings*. Stress and energy absorption were studied not only in the elastic range but also throughout the plastic range; these parameters and the types of fracture were correlated.

Mineral Industry Education Div. held a panel discussion on *Fundamental Science in Metallurgical Education*. Members of the panel were: Joseph Newton, professor of metallurgy, University of Idaho; F. R. Morral, research div., Kaiser Aluminum & Chemical Corp.; and W. M. Armstrong, professor of metallurgy, University of British Columbia. Mr. Newton presided in the absence of Panel Chairman F. A. Forward. Members of the panel and the audience were in general agreement on the need for adequate training in fundamental sciences. It was also agreed that the humanities were desirable where they could be conveniently included in the curriculum. The feeling was expressed that definite steps must be taken to correlate basic science courses with engineering courses.

The discussion led to the vanishing enrollment in mineral science courses. A rather pessimistic note was reached in this latter discussion when several persons expressed the opinion that discussion of curricula was pointless in view of the fact that it appeared likely there would be no students taking the courses in a few years.

Plans are being made and government permission sought to build a pipeline to the West Coast from the Peace River Gas Fields in northeastern British Columbia and western Alberta, according to H. R. Seykota, production sales manager, Portland Gas & Coke Co., in his paper *Natural Gas for Pacific Northwest*, presented on the Industrial Minerals program. Mr. Seykota pointed out that the Pacific Northwest produces coal, coke, wood, gas, and hydroelectric energy. It imports petroleum fuels from California.

Reopening of the Wilkeson Property in Pierce County, Wash. to Produce Metallurgical Coking Coal, by Earl R. McMillan, mine production engineer, Northwest Improvement Co., was read by H. F. Yancey, U. S. Bureau of Mines.

Local arrangements Committee Members were: Registration, Joseph Daniels; Finance, H. F. Yancey; Publicity, E. R. McMillan; Programs, L. H. McGuire; Students, D. A. Pifer; and Plant Visits, F. F. Aplan.

James T. Gow, Electric Steel Foundry Co.; W. C. Leonard, American Smelting & Refining Co.; W. W. Wiltschko, Aluminum Co. of America; Joseph Newton, University of Idaho; and Milnor Roberts, consulting engineer aided in recording events and sessions of the conference.

Next year's Pacific Northwest Conference will take place in Portland, Ore.

F. W. Libbey, director of the Oregon State Dept. of Geology and Mineral Industries, and an AIME Director, left; Ernest O. Kirkendall, center; and Milnor Roberts, dean Emeritus, University of Washington, discuss last minute details of the Northwest Conference. Mr. Roberts served as Chairman of the Industrial Minerals technical session.

RESEARCH IN PROGRESS

Research personnel are reminded of the Institute of Metals Div. Research in Progress session planned for the Fall Meeting in Cleveland. Abstracts of proposed papers not exceeding 200 words in length are to be submitted to Institute headquarters prior to Aug. 1, 1953, for inclusion in the September issue of JOURNAL OF METALS. Abstracts not received in time for publication in the September issue may, nevertheless, be presented if received prior to Sept. 15, 1953, according to B. L. Averbach, IMD Program Committee.

The Program Committee urges those interested to avail themselves of the opportunity of participation in this program in order that new research information be presented for discussion.

Powder Metal Group, Detroit Section Meet

Aims and functions of the newly formed Detroit Powder Metal Group were explained in a joint meeting with the Detroit Section of the AIME held in the Junior Room of the Engineering Society of Detroit.

The Detroit Powder Metal Group consists of a number of persons interested in advancing the technology of powder metallurgy. While all officers of the group are members of the AIME and the organization is affiliated with the Institute, membership is not limited to AIME Members.

Group officers are: J. H. Smith, Glidden Co., Chairman; P. V. Schneider, Ford Motor Co., Secretary; Henry F. Latva, Detroit Sintered Metal Corp., Treasurer; Eric W. Weinman, General Motors Corp., Vice-Chairman and Program Chairman; and John Haller, Michigan Powdered Metal Products, John H. Speck, Amplex Mfg. Co., and Harold L. Anderson, Ford Motor Co., Executive Committee.

Members of the Program Committee in addition to Mr. Weinman are: Harold L. Anderson; Carl Wiedman, Carboloy Dept., General Electric Co.; H. Gardner; L. W. Alexander, Michigan Powdered Metal Products; John S. Check, Amplex Mfg. Co.; Dalton A. Pearson, Allegheny Ludlum Steel Corp.; and Robert E. Mahr, Chrysler Jet Engine Plant. John Olson of Chrysler Engineering is in charge of membership.

New York Section Holds Joint Meeting

A joint meeting of the AIME New York Powder Metallurgy and Physical Metallurgy Groups was held May 20, 1953 at the Brass Rail in New York City. John T. Norton, MIT professor, moderated the panel discussion. R. P. Seelig, Chromalloy Corp., was meeting Chairman.

Powder metallurgy contingent on the panel posing questions for the physical metallurgists to answer were: H. H. Hausner, Sylvania Electric Products, Inc.; R. Steinitz and W. J. Leszynski, American Electro-Metal Corp.; L. L. Seigle, Sylvania Electric; and E. S. Machlin, Columbia University.

R. Steinitz posed the question of the unusual properties of sintered aluminum powder (SAP). The second problem presented was an inquiry into the effects of sintering of fine and coarse powders by Mr. Hausner. The last question was presented by Mr. Leszynski, who asked for an explanation of the apparent anomaly in sintering of a mixture of metal powders such as Cu-Ni. Others joining in the discussions were C. G. Goetzel, F. V. Lenel, J. F. Kuzmick, R. P. Seelig, H. S. Kalish, J. Adams, F. D. Rosi.

Annual Party Held By Connecticut Section

Dinner, good fellowship, and fun for all capped off the successful season of the Connecticut Section of AIME, as the group held its annual party at the Copper Kettle, Wolcott, Conn., May 15, 1953.

The entire Section membership and their ladies turned out. Many of the ladies are members of the WAAIME. Dinner followed cocktails —and there were no formal speeches.

Section Chairman L. W. Thelin greeted the guests and in his brief and informal remarks noted that the Section reached its yearly quota of new members.

Novel feature of the evening was a mystery auction, with Lloyd Raymond wielding the gavel over gaily wrapped packages of undisclosed content. The articles were contributed by firms whose engineers are members of the Section. The auction proved to be entertaining to buyers and onlookers—and also a boost for the Section treasury.

A magic show and dancing completed the evening. Members of the Party Committee were: Leon Thelin, Del Trout, Lloyd Raymond, and Lew Thelin.

T. B. Counselman Elected Vice-President

At the May 13 meeting of the AIME Board of Directors in Tulsa, T. B. Counselman was elected a Vice-President for a one-year term to expire in February 1954, to fill the unexpired term of L. F. Reinartz as Vice-President. Mr. Counselman has also been nominated for the same office for a three-year term expiring in February 1957.

Revise Bylaws For Life Membership

By paying 20 times the current annual fee, a Member or Associate Member of the Institute can become a Life Member. This was provided by a revision of Art. II, Sec. 3, of the AIME bylaws passed by the Board. Heretofore the fee had been fixed at $300 when the dues were $15. The revised bylaw now reads as follows:

Any Member or Associate Member may become, by payment at one time of 20 times the current normal annual dues for the grade in which he is enrolled, a Life Member or Life Associate Member, according to his current status at the time application is made. He shall not be liable thereafter to pay annual dues.

Reduce Local Section Appropriations

Following instructions from the Board of Directors, as agreed upon at its meeting on Feb. 17, 1952 that the Committee on Local Section Appropriations "give more careful study to the reports of Local Sections and that their actual need for money be taken into consideration in establishing the amount of individual appropriations," the Committee this year reduced the total amount recommended to be appropriated from $10,285 in 1952 to $7175. Requests from the Sections had totaled $12,175. Individual requests were cut down for all except six Local Sections.

Protests from many of the Local Sections affected were considered by the Committee (Gail F. Moulton, Chairman; Plato Malozemoff and Leo F. Reinartz) and there was lengthy discussion of the whole matter at the Tulsa meeting of the Board. It was finally voted to send out checks to the Local Section treasurers as recommended by the Committee but to invite each Section to offer further data supporting its request for a larger contribution if it felt that definite financial hardship would be suffered. Some revision of the Committee's recommendations was expected at the next Board meeting. As to future appropriations, the Board feels that the Council of Section Delegates should study the whole subject of Local Section appropriations thoroughly and make a recommendation to the Board before appropriations for 1954 are determined. There are two schools of thought among Institute members on Local Section support from the national treasury: One, that support should be continued on approximately the same scale as in the past; and the other that Local Sections should be self-supporting as far as possible, the exception chiefly being newly organized Sections and those in areas where special conditions exist.

ANNUAL MEETING PAPER DEADLINE

Sept. 15, 1953 is the deadline for all Institute of Metals Div. papers for the 1954 New York Annual Meeting and the deadline for all Iron and Steel Div., and Extractive Metallurgy Div. papers to be prepublished.

Personals

Roy W. Sidbury has been appointed district manager of the Baltimore branch of Chase Brass & Copper Co., Inc.

Frank G. Breyer was the recipient of an honorary degree of Doctor of Engineering from Clarkson College of Technology. Mr. Breyer is a partner of Singmaster & Breyer, New York.

Erhardt C. Koerper was appointed works manager of Ampco Metal, Inc., Milwaukee.

Joel Hunter has been elected a director and executive vice-president of the Crucible Steel Co. of America. He was formerly vice-president in charge of finance.

George D. Kneip, Jr., formerly with S. K. Wellman Co., Bedford, Ohio, has recently been appointed to the staff of the Oak Ridge National Laboratory, Oak Ridge.

Robert F. Smith, vice-president of the Indiana Steel Products Co., Valparaiso, Ind., and acting chief executive of the company, has been elected president and a member of the board of directors.

Martin H. Binstock has joined Sylvania Electric Products, Inc., as engineer in charge, atomic energy div., Bayside, L. I., N. Y. He had been associated with Westinghouse Electric Corp., East Pittsburgh.

Robert L. Reynolds is now metallurgical assistant, Kaiser Steel Corp., Fontana, Calif. He had been flotation operator for the Magma Copper Co., Superior, Ariz.

William J. Greene was appointed assistant director of metallurgical research for the Air Reduction Co., Inc., Murray Hill, N. J. Mr. Greene joined Air Reduction in 1946 as a research and development engineer.

James H. Potter has been named professor of mechanical engineering and associate dean at Stevens Institute of Technology, Hoboken, N. J. He will serve as head of the mechanical engineering dept. Dr. Potter had been professor of mechanical engineering at the University of Illinois.

D. R. Strouse has been appointed production manager, Tube Reducing Corp., Wallington, N. J. Prior to joining the Tube Reducing Corp., Mr. Strouse was head of production control at Morse Twist Drill & Machine Co., New Bedford, Mass.

Dean B. Valentine has been named assistant superintendent of industrial engineering, Colorado Fuel & Iron Corp., Pueblo, Colo.

E. K. MILLER

E. K. Miller, assistant vice-president, production, Jones & Laughlin Steel Corp., has been appointed vice-president, production. **John E. Timberlake** has been named vice-president, sales.

Earle R. Marble, Jr., has been promoted to assistant manager of technical operations, Federated Metals div., American Smelting & Refining Co., Newark, N. J. He had been chief metallurgical engineer.

L. E. Grubb, former general superintendent of International Nickel Co.'s Bayonne works, has been appointed general superintendent of the Huntington, W. Va. works. **P. H. Flynn,** who had been assistant superintendent at Bayonne, was named general superintendent succeeding Mr. Grubb.

Francis B. Herlihy, formerly assistant chief metallurgist, has been appointed chief metallurgist for the American Brake Shoe Co., Mahwah, N. J.

R. W. Stetson was appointed vice-president, Stuart Steel Protection Corp., Kenilworth, N. J.

Joseph L. Geenens, formerly superintendent of the Alnico permanent magnet section of Carboloy, Schenectady, has been appointed manufacturing superintendent of the Edmore, Mich., plant of Carboloy dept., General Electric Co.

A. W. Schlechten, chairman, dept. of metallurgical engineering and professor of metallurgical engineering and **J. Bruce Clemmer,** chief, Salt Lake City station, U. S. Bureau of Mines, represented the United States at a series of metallurgical conferences held in Europe by the Organization for European Economic Cooperation.

Benjamin F. Fairless, chairman of the board and chief executive officer, U. S. Steel Corp., has been awarded the Vermilye Medal by the Franklin Institute.

Frank F. Argust was appointed superintendent of industrial engineering of the Colorado Fuel & Iron Corp., Pueblo.

Charles B. Harding was elected a director of Cerro de Pasco Corp., New York. Mr. Harding is senior partner of Smith, Barney & Co.

James A. Roemer and **John J. Kraus** were promoted to vice-presidents of Sharon Steel Corp., Sharon, Pa.

Raymond L. Witzke has been appointed manager of the industrial atomic power section, Westinghouse Electric Corp., Pittsburgh.

Arthur Edward Flynn was named President-Elect, 1953 of the Canadian Institute of Mining and Metallurgy. Professor Flynn has held the chair of mining engineering at the Nova Scotia Technical College since 1921.

Emil H. Praeger has been selected Metropolitan Engineer of the Year by the Metropolitan Section of the ASCE. Mr. Praeger, a principal in the New York consulting engineering firm of Praeger & McGuire, formerly was head of the dept. of civil engineering at Rensselaer Polytechnic Institute, Troy, N. Y.

R. Genders of London, England, has been appointed metallurgical consultant to Vanadium Corp. of America. Dr. Genders is deputy director of metallurgical research at the Royal Ordnance Factory, Woolwich.

G. P. Contractor, who has been acting director of the National Metallurgical Laboratory of India is resigning from the Indian Council of Scientific and Industrial Research. He will be leaving India this summer on his way to Canada.

Richard J. Menze has been promoted to chief metallurgist at the Malvern mine and mill of Magnet Cove Barium Corp.

K. C. Brownell, American Smelting & Refining Co., New York; **E. R. Dondorf,** National Lead Co., New York; **R. E. Dwyer,** Anaconda Copper Mining Co., New York; **Andrew Fletcher,** St. Joseph Lead Co., New York; **Elmer Isern,** Eagle-Picher Co., Miami, Okla.; **G. H. LeFevre,** U. S. Smelting, Refining & Mining Co., New York; **W. H. Leverett,** National Zinc Co., Inc., New York; **E. H. Snyder,** Combined Metals Reduction Co., Salt Lake City, Utah; **S. D.**

Strauss, American Smelting & Refining Co., New York; and **Jean Vuillequez,** the American Metal Co., Ltd., New York, were elected directors of the American Zinc Institute.

Preston C. Mitchell has been elected vice-president, Harbison-Walker Refractories Co. **Edward T. Hill** and **Harry H. Yaeger** have been elected directors.

Edward N. Chilson has been elected chairman of the board and **Ford Kurtz,** president, J. G. White Engineering Co., New York.

H. W. Franz is plant manager for Colonial Aluminum Smelting Corp., Columbia, Pa.

E. J. Walsh is now executive director of the Foundry Educational Foundation, Clevland.

Albert J. Phillips, director of the research dept., was elected vice-president of American Smelting & Refining Co.

Obituaries

Bertram D. Quarrie (Member 1908) died in Cleveland, Ohio on Feb. 15, 1953, according to Institute records. Mr. Quarrie was born in Cleveland and graduated from Case. He received a B.S. in 1901 and an E.M. in 1904. He was employed as a chemist by various firms and in 1905 was made superintendent, Cleveland Furnace Co. He was promoted to assistant superintendent, central works in 1906. Mr. Quarrie joined the Inland Steel Co. as superintendent of furnaces in 1907. In 1908 he was general superintendent for Newburgh Steel Works, Cleveland. He became general manager for the Otis Steel Co. in 1923 and was president of the Oliver Iron & Steel Corp., Pittsburgh. Mr. Quarrie was president of the Cuyahoga Valley Waterworks, Cleveland in 1933.

Francis M. Walters, Jr.

An Appreciation by

Frank T. Sisco

The science of physical metallurgy lost one of its strongest proponents, and the art of precise metallurgical research lost one of its most skilled exponents, when Francis M. Walters, Jr., died at Los Alamos, N. Mex., on April 18, just seven days before his 65th birthday. Frank Walters was born at Monticello, Ind., on Apr. 25, 1888. His undergraduate and graduate work, all of it in physics, was done at the University of Missouri, where he received his A.B., A.M., and Ph.D. degrees. After a term of teaching physics, between his master's and doctor's degrees, he entered that superb training school for young scientists—the National Bureau of Standards—where he stayed for four years, leaving in 1924 to become director of the Bureau of Metallurgical Research (later the Metals Research Laboratory) at Carnegie Institute of Technology.

It was while Frank Walters was at Carnegie Tech that he and his associates, Maxwell Gensamer, Cyril Wells, J. F. Eckel, and V. N. Krivobok, all well-known names in present-day metallurgy, carried on the research work on the iron-manganese-carbon system that won Frank Walters and these co-workers the Henry Marion Howe gold medal of the American Society for Metals.

It is unnecessary to list Dr. Walter's accomplishments from the time he left Pittsburgh until he accepted, in 1946, a highly important research post at the Los Alamos Scientific Laboratories, on that picturesque and renowned mesa, thirty miles from Santa Fe. These are given in *Who's Who in America, American Men of Science,* and *Who's Who in Engineering.* His last public honor before he disappeared behind the high fence of the "classified technical area on the Hill," where the atomic bomb is made, was the Navy Distinguished Civilian Award, presented to him in 1946 for his work as superintendent of the metallurgy div. at the Naval Research Laboratory in Washington.

We have known Frank Walters intimately for nearly 25 years—ever since his Carnegie Tech days, when the Engineering Foundation and Alloys of Iron Research were supporting his research work on the iron-manganese-carbon system—and all during that time we have admired him tremendously for his intellectual honesty as well as for his scientific and research talents, and for his sympathetic understanding of the tyro scientist and a willingness to help many a young man to become adjusted to a sometimes difficult environment.

Fundamentally, Frank Walters was shy and unassuming; but he could be impatient and forthrightly critical when conclusions were drawn and theories formulated as the result of an extrapolation from too few experimental data. He was more than ordinarily happy in his home life; for 37 years he shared his problems and achievements with a wife who complemented his personality. His three sons are a credit to their parents.

Everyone who knew Frank Walters will miss him.

NECROLOGY

Date Elected	Name	Date of Death
1904	Clarence E. Abbott	Apr. 14, 1953
1951	Harry Dobrin	October 1952
1898	Harlan S. Emlaw	Feb. 5, 1953
1922	Elmer H. Finch	Apr. 29, 1953
1920	R. E. Gruber	January 1953
1939	Willard M. Hall	Apr. 9, 1953
1942	B. C. Leadbetter	Oct. 10, 1952
1916	Charles Lindmueller	May 3, 1953
1931	Sture Mortsell	1951
1931	Francis M. Walters, Jr.	Apr. 18, 1953

Proposed for Membership — Metals Branch AIME —

Total AIME membership on May 31, 1953 was 18,822; in addition 1227 Student Associates were enrolled.

ADMISSIONS COMMITTEE

O. B. J. Fraser, Chairman; Philip D. Wilson, Vice-Chairman; F. A. Ayer, A. C. Brinker, R. H. Dickson, Max Gensamer, Ivan A. Given, Fred W. Hanson, T. D. Jones, G. W. Lutjen, E. A. Prentis, Sidney Rolle, J. T. Sherman, F. T. Sisco, R. L. Ziegfeld.

The Institute desires to extend its privileges to every person to whom it can be of service, but does not desire as members persons who are unqualified. Institute members are urged to review this list as soon as possible and immediately to inform the Secretary's office if names of people are found who are known to be unqualified for AIME membership.

In the following list C/S means change of status; R, reinstatement; M, Member; J, Junior Member; A, Associate Member; S, Student Associate.

California
San Francisco—Gleason, David S. (J) (R. C/S—S-J)
San Francisco—Vorell, Frank J. (J) (R. C/S—S-J)

Colorado
Denver—Franz, Alwin F. (M)
Golden—Cook, Douglas R. (J)

Connecticut
New Canaan—Monroe, Eugene H. (M) (R. C/S—J-M)
Old Greenwich—O'Mara, Fred B. (A)
Old Greenwich—Wemple, Edward L. (M)
Watertown—Manning, Edward M. (M) (R. M)

Delaware
Claymont—Bruce, George W. (M)
Wilmington—Gentry, Roger W. H. (A)

Illinois
Chicago—Gamson, Bernard W. (M)
Chicago—McCosh, William C. (A)
Chicago—McCurdy, Charles J. (M)
Chicago—Powell, Harry H. (M)
Chicago—Riley, Lawrence R. (M)
Chicago—Ziomek, Henry A. (A)
Urbana—Ang, Choh-Yi (A) (R. C/S—A)

Indiana
Chesterton—Gatewood, Emmett H. (J)

Missouri
St. Louis—Trinkle, Arthur H. (M)

New Jersey
Union City—Berry, Thomas F. (M)

New York
Floral Park—Hydrean, Peter P. (M) (C/S—A-M)
New York—Fraenkel, Ernest (A)
New York—Ornstein, Jacob L. (J) (R. C/S—S-J)
Schenectady—Baldwin, Elmer E. (M) (R. C/S—A-M)

Ohio
Canton—Wyandt, John J. (M)
Cleveland—Steudel, George E. (M)
Columbus—Marsh, Lyle L., Jr. (J)
Lakewood—Heilig, Charles R. (A)
Lakewood—Matz, Kenneth R. (M)
No. Kenova—Horsley, Amos F. (M)

Pennsylvania
Palmerton—Colton, Nelson B. (J)
Pittsburgh—Gaebe, Philip A. (M)
Pittsburgh—Pottmeyer, Edward W. (M)
Sewickley—White, John H., Jr. (M)
White Haven—Moser, Bernard A. (M)

South Carolina
Aiken—Winans, William B. S. (M)

Texas
El Paso—Holcombe, William I. (M)

Chile
Concepcion—Chavez, Servando (M) (R. M)

Norway
Sauda—Blegen, Harald M. (M)
Sauda—Eide, Cato (M)

Nova Scotia
Sydney Forks—Dean, Alfred W. (M) (R. M)

Ontario
Burlington—Weir, William R. (M)
Sault Ste. Marie—Hargreaves, Charles G. (M)

Peru
Lima—Ballon, Alfonso (M) (C/S—A-M)

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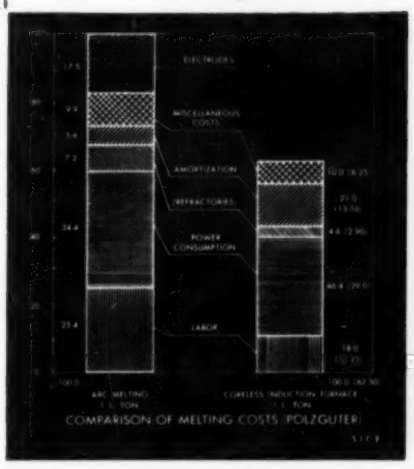
▼ Comparison of Melting Costs



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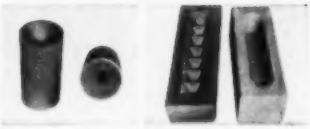
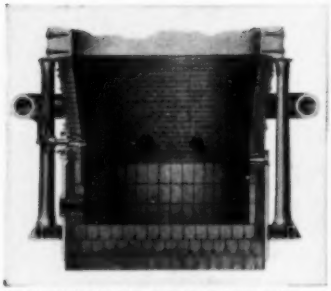
GRAPHITE CRUCIBLES



GRAPHITE MOLDS

GRAPHITE SINTERING BOATS & TRAYS



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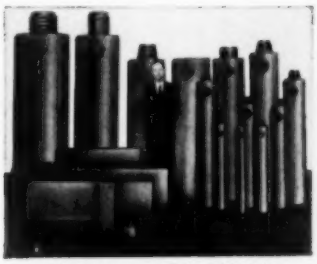
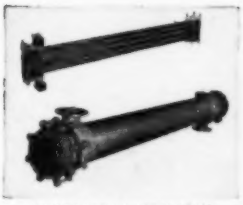


CARBON & GRAPHITE ELECTRIC FURNACE ELECTRODES

GRAPHITE FLUXING TUBES



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